THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

JULY, 1958

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Production of High-Grade Iron Ore at International Nickel Company's Plant

P. QUENEAU, E. H. BRACKEN and D. KELLY: 'Production of High-Grade Iron Ore at Copper Cliff, Ontario.' *Amer. Inst. Mining, Metallurgical and Petroleum Engineers, Preprint*, Feb. 1958; 16 pp.

Ore milled by The International Nickel Company of Canada, Ltd., in the Sudbury District of Ontario, consists essentially of pentlandite, nickeliferous pyrrhotite, chalcopyrite and rock minerals. Until recent years the sulphide minerals were recovered by flotation in a mixed pentlandite-pyrrhotite nickel concentrate and a copper concentrate. Since the iron : nickel ratio of the nickel concentrate was approximately 10 : 1, the controlling factor in nickel-smelting capacity was the large quantity of iron to be slagged. Studies on separate treatment of nickeliferous pyrrhotite concentrate for iron recovery indicated that this could be achieved on a satisfactory basis through development of economical procedures whereby: (*a*) the nickel content of the concentrate could be reduced to below 1·0 per cent., with accompanying virtual elimination of copper, precious metals and silicate contamination; (*b*) the sulphur content of the concentrate could be reduced to below 1·0 per cent., while producing a gas having a sulphur-dioxide content of 10 per cent. or more; (*c*) the nickel content of the resulting iron oxide could be reduced to 0·15 per cent. or less. Requirement (*a*) was met many years ago, but it was only in 1954 that the development of methods which would satisfy requirements (*b*) and (*c*) was sufficiently advanced to justify commercial operation.

The laboratory and pilot-plant stages of the INCO Iron Ore Recovery Process were described in a paper presented to the Canadian Institute of Mining and Metallurgy in 1956. The present paper records details of commercial operation of the process, at the Copper Cliff plant of the Company.

A plant having a capacity for treatment of 365,000 tons of pyrrhotite per annum started operation early in 1956, as the first unit of an installation designed to provide eventually an annual capacity of more than 1,000,000 tons.

Elimination of sulphur is accomplished in fluid-bed roasters operating in closed circuit with cooling boilers and cyclones. Nickel is extracted by atmospheric-pressure leaching in ammoniacal solutions, following selective reduction of the calcine in kilns using concurrent gas-solids flow.

Permanent-magnet equipment is used for solid-liquid separation in the leaching operation and steam kettles are employed for nickel precipitation from pregnant solution. The magnetite fines are agglomerated into one-inch balls by pelletizing on discs and firing on a travelling grate.

Each of the operations involved in the process is described, with illustrative flow sheets.

Over 200,000 long tons of 68 per cent. iron pellets containing phosphorus 0·003, SiO_2 1·5, sulphur 0·01, nickel 0·15, per cent., had been produced to the date of this paper.

The product, which is believed to be the highest-quality iron ore produced in quantity in North America, is being successfully used, as open-hearth charge ore, by the steel industry in Canada and the U.S.A.

NICKEL

Influence of Texture on Physical Properties of Nickel Sheet

E. R. W. JONES, C. A. CLARK and E. A. FELL: 'Calculated and Observed Effects of Texture on the Magnetic Properties and Young's Modulus of Nickel Sheet.' *Brit. Jnl. Applied Physics*, 1958, vol. 9, May, pp. 178–84.

It is well established that the magnetic domains in annealed ferromagnetic materials are aligned as far as possible along preferred crystallographic directions. Practical application of this effect is illustrated by the deliberate production of cube texture in 3 per cent. silicon-iron and 50 per cent. nickel-iron alloys. The cubic orientation may be produced in face-centred-cubic metals by a heavy cold reduction and subsequent high-temperature anneal, the annealing temperature for best results often being rather critical. In iron, 3 per cent. silicon-iron alloy and 50 per cent. nickel-iron alloy, the cube edge is the easy direction of magnetization and the production of a cube texture improves the maximum permeability of the material.

Although the roll and recrystallization textures for face-centred cubic metals generally have been studied by several investigators, little work has yet

been done on the dependence of magnetic properties on texture in sheet material for which the ⟨ 111 ⟩ direction is the easy direction of magnetization, as in nickel. The work now described was designed to assist in filling the gap. It comprised study of the processing and annealing treatments required for production of nickel strip, with and without texture, and determination of the average magnetic properties of the materials, especially the initial magnetization curve, the hysteresis loop, the electromechanical coupling coefficient and Young's modulus.

Experimental values are recorded for high-purity crystalline nickel sheet produced by powder metallurgy and annealed in purified hydrogen. Comparison between theory and experiment indicates good agreement for Young's modulus and fair agreement for magnetic properties. The textures were determined by X-ray photographic techniques and it was shown that strip possessing a cube recrystallized texture gave lower properties than recrystallized strip having a random grain orientation. It was also found, in agreement with previous work on copper, that the perfection of the cube texture was governed mainly by the temperature of the annealing treatment.

Chemisorption of Hydrogen on Nickel

L. VASKA and P. W. SELWOOD: 'The Mechanism of Chemisorption: Hydrogen on Nickel at Elevated Pressures.'

Jnl. Amer. Chemical Soc., 1958, vol. 80, Mar. 20, pp. 1331–5.

This paper is the third in a series reporting studies of the mechanism of chemisorption, which were made by measuring changes in magnetization occurring during chemisorption of the gas on supported nickel catalysts of various types. (Abstracts of previous papers appeared in *Nickel Bulletin*, 1956, vol. 29, No. 10–11, p. 181; 1958, vol. 31, No. 1, p. 3.) The work now published extends the investigation to measurement of hydrogen adsorption on a silica-supported nickel catalyst at pressures up to 140 atmospheres, and correlation of the measurements with decrease in magnetization.

Details are given of the volumetric method used to make simultaneous measurements of adsorption and specific magnetization. Results are given in terms of magnetization-pressure and magnetization-volume isotherms, all at room temperature.

The principal observation made was that hydrogen adsorption at progressively increasing pressures caused a progressive decrease in magnetization of nickel. The mechanism of this phenomenon is discussed. The authors summarize their conclusions as follows:

'The results of these studies demonstrated that the monolayer of chemisorbed hydrogen on the surface of nickel in the silica-supported catalyst is not complete at 1 atm., but, for all practical purposes, the saturation is reached at about 100 atm. It may be estimated that the increase of instantaneous chemisorption caused by increasing the pressure from 100 to 200 atm. would be only about 0·1 cc./g. We have not developed direct evidence concerning possible penetration of hydrogen into the nickel lattice at high pressures, but the fact that the data can be represented by the Langmuir monolayer equation, together with arguments advanced by Doerner, all tends to show that if such penetration occurs it must be an extremely slow process.'

Change in magnetization of nickel is regarded as a definite and direct measure of the quantity of hydrogen chemisorbed.

Adsorption and Diffusion of Hydrogen on Nickel

R. WORTMAN, R. GOMER and R. LUNDY: 'Adsorption and Diffusion of Hydrogen on Nickel.'

Jnl. Chemical Physics, 1957, vol. 27, Nov., pp. 1099–107.

Study of surface diffusion of hydrogen on tungsten had shown that migration depended markedly on the topography of the surface, and that much could be learned about the latter aspect by diffusion experiments. The work now reported was undertaken to compare the behaviour of hydrogen on a body-centred-cubic substrate with that observed on a face-centred-cubic base, since the structures of the two vary widely. Nickel was peculiarly suitable for the comparative investigation because its lattice constant is similar to that of tungsten and because the heats of binding of hydrogen atoms on nickel and on tungsten are comparable.

It was found that, in nickel, diffusion occurs with an activation energy of 7 ± 1 kcal in the chemisorbed layer, without the precipitation boundaries observed in the case of tungsten. This difference is believed to result from the inherent homogeneity of f.c.c. substrates, on which most adsorption sites appear to be equivalent. This conclusion is supported by the marked lack of emission anisotropy of hydrogen-covered nickel field emitters. A field-dependent transformation in the adsorbed film was found between 2° and 4°K. (−271° and −269°C.): it is interpreted as a shift in the equilibrium between adsorbed hydrogen molecules and the terminal fraction of adsorbed hydrogen atoms. The implications of this observation, in relation to the mechanism of hydrogen–deuterium exchange, are discussed.

Diffusion between Uranium and Nickel

C. L. ANGERMAN: 'Electron Metallography of the U-Ni Diffusion Zones.'

Nuclear Engineering and Science Conference, Chicago, Mar. 1958, *Preprint* 63, *Session IX*; 6 pp. + tables and figures.

The microstructures of the intermediate compounds formed by diffusion in couples of wrought uranium and nickel and of wrought uranium and electrodeposited nickel were studied by electron metallography and X-ray-diffraction techniques. The

solution used for deposition of nickel on uranium was of the following composition:

	oz./gal.	*g./L.*
$NiSO_4.6H_2O$	13	97·5
$NaSO_4.7H_2O$	13	97·5
NH_4Cl	4	30
H_3BO_4	2	15

A current density of 16 amp./ft.^{2} (1·7 amp./dm.^{2}) was used; the pH of the solution was 5·3–5·8, and the temperature of operation was 35°C. The coatings deposited were 0·0005–0·0015 in. (0·0125–0·0375 mm.) thick.

As would be predicted by the nickel–uranium phase diagram, seven intermetallic compounds were observed. The conditions in which they were formed are described and typical structures are illustrated.

Thermodynamics of Nickel/Solution Systems

K. MÜLLER: 'The Electrochemical Thermodynamics of the Nickel/Solution System.'

Metalloberfläche, 1958, vol. 12, May, pp. 133–6.

After reviewing the formulae generally employed to express thermodynamic reactions involved in metal/solution systems, the author exemplifies their use by a description (supplemented by graphs) of 15 thermodynamic electrode reactions which can occur in nickel/solution systems. By way of illustrating the practical value of these theoretical considerations, the graph is then applied to indicate the probable behaviour of nickel in a specific solution.

Volumetric Determination of Nickel in Catalysts

O. N. DOBRYNINA and K. G. BOGAREVA: 'Volumetric Method for Determination of Nickel in Catalyst Powders.'

Masloboino-Zhirovaya Prom., 1957, vol. 23, No. 9, p. 40.

The following method is recommended:

'Dissolve 2 g. of powdered sample in dilute HCl (1 : 5); heat to boiling; cool; transfer solution to 250-ml. volumetric flask; dilute with distilled H_2O, mix, and allow to stand until settled. Place 10 ml. of clear supernatant or filtrate in a 250-ml. flask, add 30 ml. distilled H_2O, and 20 ml. of dilute NH_4OH (1 : 5), and wait until blue colour develops. Add dry murexide indicator and titrate resulting muddy-green solution with 0·1-*N* 'Trilon B' solution until colour changes from green to violet. Then *X* (nickel content of powder) = *Ta*250(100)/10(2), where *T* represents titre of 'Trilon B' solution against nickel standard solution, and *a* = ml. of 'Trilon B' solution used.'

Polarographic Analysis of Electronic Nickel

'A New Analysis for Nickel Cathodes.'

Bell Laboratories Record, 1958, vol. 36, Apr., pp. 146–7.

In view of the well-known influence of trace elements on the behaviour and life of thermionic cathodes, analytical methods of high precision must be used for assessment of quality of the nickel used. The most rapid technique consists in spectrographic determination of the impurities present, but in the course of research designed to make possible even higher accuracy than can be secured by such procedure, chemists in the Bell Telephone Laboratories have applied the method of polarography. It has been found that it is thus possible to improve the average accuracy of the results from the five per cent. level obtainable with spectrographic analysis to one–two per cent. A further advantage of the polarographic method is that no standards similar to the sample are required. Each impurity is determined independently, and the presence or absence of another element has no influence on any individual estimation. It is noted that a novel feature in the polarographic determination of iron and manganese in the nickel comprises the addition of triethanolamine to solutions containing these two elements. Such an addition results in improved constant current and, in turn, in clearer definition of the polarographic wave representing the ions. The broad principles of both spectrographic and polarographic methods of analysis are outlined in this note.

Use of Nickel for Handling Fluorine and its Compounds

See abstract on p. 198.

Separation of High-Grade Iron Ore in Nickel-Extraction Process

See abstract on p. 183.

ELECTRODEPOSITION AND OTHER COATING METHODS

Levelling in Nickel-Plating Solutions

E. RAUB: 'Research on Microthrowing Power and Levelling of Plating Baths.'

Plating, 1958, vol. 45, May, pp. 486–92.

The paper opens with a brief survey of the conclusions drawn by other investigators with regard to the factors influencing levelling and the mechanism of the smoothing action. The experimental work described consisted in study of the distribution of deposited copper and nickel on the surfaces of microprofiles consisting of cylindrical test pieces closed at one end, onto which metal was electrodeposited from agitated and from still electrolytes. The test profile is illustrated. In investigating the influence of the shape of surface profile on microthrowing power, the cathodes used were in the form of plates having V-shaped notches (the angle of which varied from 30° to 120°, with depth of notch ranging from 0·1 to 0·14 mm.) and U-shaped notches.

In plating the closed-end cylindrical test pieces two nickel solutions were used: a 'good levelling bath of proprietary composition' and a Watts solution

containing (g./L.) nickel sulphate ($NiSO_4.7H_2O$) 240, potassium chloride 20, boric acid 30. Operation was at 55°C., with a cathode current density of 3–5 amp./dm.^{2} The average thickness of the nickel coatings was 10–40 microns (0·4–1·6 mils). The copper solution was of the cyanide type and a thickness of 10–30 microns (0·4–1·2 mils) was deposited. For the experiments in which notched-plate brass panels were used, the types of solution employed were similar to those mentioned above, and, in addition, a Watts bath containing the sodium salt of alpha-naphthalene sulphonic acid was employed. (Such a solution is known to exert no levelling action.) For this group of tests a copper-sulphate, as well as the copper-cyanide, solution was used.

The results obtained are discussed at length, on the basis of illustrative charts and of photomicrographs demonstrating the structure of deposits made under the various conditions used.

The following broad conclusions are drawn:

There is a fluid transition between macro- and micro-throwing power. In deposition of copper from a cyanide solution the characteristic features of micro-throwing power show themselves also on macro-profiles if the form of the latter is such as to exert a strongly repressing effect on convection of the electrolytes, so that a thick diffusion layer forms in the depressions.

Levelling in the range of micro-throwing power occurs with coatings deposited from the copper-sulphate bath and the Watts nickel bath containing no special addition agents. The levelling is the result of the definite field-oriented type of crystal growth of the metal crystallizing on the sides of the micro-profiles.

On cathodes having V-shaped notches there is no concentration of the lines of current flow into the recessed section of the micro-profiles: the cathodic current density in the notches does not exceed the average value on the surface, and is in some cases smaller. There is therefore no levelling.

Genuine levelling, in the sense of forcing the current lines into the depths of the profile, occurs with bright-nickel plating solutions which contain levelling agents, but even with strongly levelling nickel-plating electrolytes the growth of the coating in the lowest portion of the profile is weaker than in the middle and upper portions, with the result that a void may be formed and subsequently covered by deposited metal. Brass panels carrying various shapes of notch showed formation of masked cavities resulting from greater growth of the nickel deposit in the upper part of the micro-profile.

Influence of the Buffer Content of the Solution in Electroless Deposition of Nickel

L. BOSDORF and K. MÜLLER: 'Investigation of Variation in the pH Value of a Solution used for Electroless Deposition of Nickel.'

Metalloberfläche, 1958, vol. 12, May, pp. 150–1.

In the deposition of nickel by the electroless process the pH value of the solution plays a decisive rôle in determining the rate at which deposition takes place and therefore the rate of growth of the deposit. The rate of deposition increases with rise in pH, but too high (or too low) a pH value can be deleterious, resulting in an abrupt fall in the deposition rate, and even leading to decomposition of the solution. Experiments have shown the optimum value to be within the limits 4·5–5, a range in which, moreover, best results are achieved from addition of buffers such as the oxymonocarbon acids and their salts. The work reported in this paper was undertaken to determine

(1) the effect of additions of such a buffer (sodium glycolate) on the pH value and rate of growth of the deposit, and

(2) the buffer concentration producing optimum results.

Operating conditions and the composition of the solution are given below. The sodium glycolate was added in amounts ranging from 10 to 60 g./L.

	g./L.
Nickel chloride	30
Cobalt acetate (added as a brightener)	2·5
Sodium hypophospite	10

Temperature 97°–98°C.
pH (at the beginning of each experiment) 4·5
Iron plates were used as anodes.

The pH of the solution and rate of growth of the deposit were determined at half-hourly intervals: the results are presented as curves giving the variation in pH and in rate of growth as a function of time and concentration of sodium glycolate.

With no buffer addition the pH of the solution was found, at the end of 120 minutes, to have fallen to 3·08, but sodium glycolate had a corrective effect, and as the amount of the buffer was increased the fall became less drastic. With an addition of 60 g./L. of sodium glycolate the curve had almost flattened out and the pH value after 2 hours was 4·31.

These results, however, showed no correlation with those gained in establishing the effect of varying sodium-glycolate additions on the rate of deposition from the solution. (The criterion for evaluation of this factor was the thickness of the nickel coating after deposition for 120 minutes.) On the basis of previous experience it had been expected that the increase in pH values resulting from increasing additions of buffer would result in a corresponding increase in the deposition rate. The curve, however, reached a maximum of 34μ at a 30 g./L. concentration of sodium glycolate, whereas at a 60 g./L. it fell to 29μ. From these data the authors conclude that the rate of deposition is governed not only by the pH value but also by the catalytic effect of the buffer. The glycolate should, therefore, be used at a concentration which achieves the optimum combination of catalytic effect and pH value, i.e., 30 g./L.

In further experiments, sodium glycolate, glycollic acid, magnesium lactate and lactic acid were each used as buffers, to determine the effect of substituting

acids for salts. In both cases addition of the salts not only achieved the higher rate of deposition but was also associated with less variation in the pH value of the solution.

Effect of Oxide Films on the Cathode

H. B. LINFORD and D. O. FEDER: 'Cleaning and Preparation of Metals prior to Electroplating. Effect of Oxide Films. X. Experimental Results.'

Plating, 1958, vol. 45, Apr., pp. 349–59.

Report issued under the aegis of A.E.S. Research Project No. 12.

Metal surfaces prepared for plating usually enter the plating solution with some degree of contamination of the surface: oxide is particularly likely to be present. A.E.S. Research Project No. 12 has the assignment of studying the effects of plating onto surfaces contaminated by oxide films and, in association with that work, is investigating the mechanism by which plating occurs on oxide-soiled surfaces. Reports already published, on various aspects of this work, are listed in the *bibliography* accompanying the present paper.

Section X covers results obtained in electrodeposition of nickel on oxidized copper. The cathodes were copper tubing, to which, after degreasing and hydrogen-reduction treatment, oxide films containing up to 2900 μg./dm.² of oxygen (equivalent to a thickness of about 4000 Å of Cu_2O) were applied. Nickel was then electrodeposited immediately (without allowing contact of the cathodes with air). The solution used was a high-pH Watts type.

From the extensive data recorded it is concluded that, when plating oxide-soiled copper surfaces, electrochemical reduction of copper oxide consumes a large proportion of the current. With relatively thin films (less than 650 Å) all the oxide present at the start of plating may be removed by this means. There appears to be little doubt, however, that, where oxide films of greater thickness are present when plating is begun, appreciable amounts of oxide remain on the cathode (presumably under the metal coating) after electroplating. The amount of the residual oxide increases with the thickness of the film initially present.

Experiments in which oxidized copper cathodes were soaked in the Watts solution prior to plating showed that, even at high pH, significant dissolution of oxide occurs, producing a cathode film which is rich in copper ions. These copper ions co-deposit with the first layer of nickel, producing an initial coating of copper-nickel alloy. Current density (varied in these experiments over the range 10–50 amp./ft.² : 1·5·4 amp./dm.²) appears to be without significant influence on any of the electrode processes mentioned above.

Diffusion between Uranium and Electrodeposited Nickel

See abstract on p. 184.

Electrodeposition of Nickel Alloys from Pyrophosphate Solutions

T. L. R. CHAR: 'Anodic Corrosion of Metals and Alloys in Pyrophosphate Solutions.'

Corrosion Prevention and Control, 1958, vol. 5, Apr., pp. 37–8.

The author has recently published a series of papers dealing with the electrodeposition of various metals and alloys from pyrophosphate solutions (see abstracts in *Nickel Bulletin*, 1957, vol. 30, No. 12, p. 218; 1958, vol. 31, No. 1, p. 4).

The present report covers studies of the behaviour, in such electrolytes; of anodes of the following materials: tin, zinc, nickel, copper, lead, tin-copper, tin-zinc, tin-nickel, tin-lead, copper-zinc and copper-nickel. The tests comprised determination of anode efficiency and potential, and, in the case of the alloy anodes, the solutions were analysed. Structures of the alloy anodes were examined by X-ray methods.

The investigations showed that the behaviour of most of the anodes was satisfactory over a wide range of operating conditions, including metal content of the solution, ratio of pyrophosphate to metal, pH, temperature, and current density. Comments on the characteristics of individual types of anode include the following notes on the nickel and tin-nickel types:

'Unless the chloride ion was present in the solution, nickel exhibited total passivity in pyrophosphate solutions . . . Only tin dissolved from the tin-nickel electrode, and the electrodeposition of tin-nickel alloys was therefore carried out with insoluble anodes. It was, however, possible to work the tin-nickel bath with tin and nickel anodes under separate control.'

NON-FERROUS ALLOYS

Welding of 90-10 Cupro-Nickel

V. ABAVARICH: 'Cupro-Nickel Welded with Aluminium Bronze.'

Welding Jnl., 1958, vol. 37, Mar., pp. 220–4.

Difficulties have been encountered in welding the 90-10 copper-nickel alloy. The thermal conductivity of this alloy is almost double that of the 70-30 type of cupro-nickel, necessitating the use of higher current densities in order to flow the weld metal effectively. When 90-10 electrodes or filler rods are used, the higher heat, employed in association with an alloy which has a tendency to hot-shortness, sometimes results in excessive cracking of the deposit.

This paper records tests made to determine the suitability of A.S.T.M. standard aluminium-bronze electrodes (ECuAl-A2 type), using the inert-gas consumable-electrode process. The conclusion drawn from the experiments is that such procedure is suitable for joining of 90-10 cupro-nickel to similar material and for welding the cupro-nickel to mild steel. The work was carried out under standard conditions, by operators having no specialized

experience in welding cupro-nickel, but no weld failures such as cracking or excessive porosity were encountered. Field and laboratory tests are being carried out to determine the resistance of such joints to various corrosive media.

Diffusion between Uranium and Nickel

See abstract on p. 184.

Nickel Alloys for Handling Fluorine and its Compounds

See abstract on p. 198.

NICKEL-IRON ALLOYS

Magnetism and Magnetic Materials: Third International Conference, 1957

The heightened interest which has recently developed in magnetism and magnetic materials is reflected in the papers presented for the 1957 Conference, which are published in *Journal of Applied Physics*, 1958, vol. 29, Mar., pp. 237-548.

The scope of the interest of the proceedings, and of the individual contributions, is briefly indicated below. The Conference was jointly sponsored by the American Institute of Electrical Engineers, in co-operation with the American Physical Society, the American Institute of Mining, Metallurgical and Petroleum Engineers, the Institute of Radio Engineers, and (in 1957) the Office of Naval Research.

The avowed design, in planning the meeting, was to attract 'papers dealing with the basic exploration of magnetic phenomena which have some bearing on potential engineering applications and with the development of apparatus and techniques which make use of recent advances in magnetism.' In the foreword to the report the comment is made that the Conference brought out how rapidly the effective interplay of physics and engineering in magnetism leads to developments which are certain to have far-reaching effects on contemporary technology. Specific examples to which attention is directed include the recent invention of the ferromagnetic microwave amplifier, the closer investigation of ferromagnetic-resonance phenomena, the significance of the rapid reversal of magnetization which occurs in switching, advances in the theory of the magnetic properties of small particles, closer study of the origin of magnetism in certain alloys, and the better understanding of ferromagnetic anisotropy in certain ferrites, on an atomic level.

The scope of the respective sessions of the Conference, with notes on papers which are relevant to nickel-containing materials, is shown below:

Introductory Session containing papers dealing with general magnetic phenomena,
including

E. KONDORSKY, O. S. GALKINA and L. A. TCHERNIKOVA: 'Nature of Electrical Resistivity of the Ferromagnetic Metals at Low Temperatures'; pp. 243-6.

Study of the resistivity of nickel, iron and nickel-copper alloys (copper up to 25 per cent.), at temperatures from 2° to 78°K. (−271° to −195°C.).

E. F. BERTAUT AND F. FORRAT: 'Structure and Ferrimagnetism of the Ilmenite Compound $MnNiO_3$'; pp. 247-8.

E. T. FERGUSON: 'Uniaxial Magnetic Anisotropy Induced in Fe-Ni Alloys by Magnetic Anneal'; pp. 252-3.

The uniaxial magnetic anisotropy induced in iron-nickel alloys by a magnetic anneal was determined as a function of the composition, annealing temperature and duration of treatment.

Magnetization Reversal and Thin Films
including

D. O. SMITH: 'Static and Dynamic Behaviour of Thin 'Permalloy' Films'; pp. 264-73.

Thin magnetic films (about 1000 Å) evaporated onto a heated glass substrate in the presence of a d.c. magnetic field develop a uniaxial magnetic anisotropy energy of the form $E_k = K \sin^2\Phi$, with Φ measured from the deposition field. In digital-computer memory systems, such films promise to outperform ferrite cores by several orders of magnitude. The authors present a detailed static and dynamic theory based on the above anisotropy energy and the Landau-Lifshitz dynamical equation.

C. D. OLSON and A. V. POHM: 'Flux Reversal in Thin Films of 82% Ni, 18% Fe'; pp. 274-82.

The magnetization-reversal process occurring in iron-nickel films (nominally 82% Ni - 18% Fe) deposited in the presence of a magnetic field to a thickness of about 1000Å to 4000 Å have been examined by the application of appropriate fields. Experimental results indicate that at least two different magnetization-reversal mechanisms are effective. The first, characterized by relatively long re-magnetization periods, involves domain-wall movement; the second, characterized by relatively short re-magnetization periods, is consistent with the rotation of the magnetization in the plane of the film. The threshold for the rotational process is altered by the application of a transverse magnetic field in a manner consistent with a simple energy model.

F. B. HUMPHREY: 'Transverse Flux Change in Soft Ferromagnetics'; pp. 284-5.

Results of an investigation of magnetic-flux change in the direction perpendicular to the direction of the applied field indicate that the maximum flux in the transverse direction does not occur at the time when the sample is half reversed. From these data, taken on thin evaporated films of iron-nickel, it is concluded that a coherent rotation theory which assumes a single magnetization vector to

represent the entire sample is not adequate to explain the results. It is suggested that when a small transverse field is present the magnetization starts to reverse by coherent rotation, but soon loses phase throughout the sample, so that the reversal is completed by non-coherent rotation.

E. N. MITCHELL: 'Effects of Heat-Treatment of Thin Ferromagnetic Films at Intermediate Temperatures'; pp. 286–7.

Nickel-iron (82–18 per cent.) films deposited *in vacuo* in the presence of a magnetic field were annealed in an inert atmosphere. The results established that when the annealing is performed in an orienting field of 30 oersteds initial magnetic properties of these films can be altered provided that the initial temperature is sufficiently high. If a field is applied at right angles to the original preferred direction and in the plane of the film, the magnitude of the magnetic anisotropy can be reduced without changing the preferred direction. The preferred direction of magnetization can be changed if the annealing temperature is sufficiently high.

R. M. SANDERS and T. D. ROSSING: 'Reversible Rotation in Magnetic Films'; pp. 288–9.

The report is concerned with the reversible rotation of the magnetization vector of 'Permalloy' films under the influence of a magnetic field transverse to the easy direction of magnetization, and detection of the flux change parallel to the easy direction.

D. O. SMITH and G. P. WEISS: 'Steady-State and Pulse-Measurement Techniques for Thin Magnetic Films in the vhf-uhf Range'; pp. 290–1.

A v.h.f.-u.h.f. bridge consisting of two loops placed symmetrically in a rectangular coaxial cavity was used to study steady-state and pulse relaxation in thin 'Permalloy' films (about 1000 Å).

M. H. SEAVEY and P. E. TANNENWALD: 'Ferromagnetic Resonance in Ultra-Thin Films'; pp. 292–3.

A ferromagnetic film may be arbitrarily considered as 'ultra-thin' when its thickness is less than 100 Å. The author reports that it has been found possible to observe microwave resonance absorption in 'Permalloy' (80–20) films down to approximately 15 Å thickness. The magnetization drops sharply near 60 Å, and ferromagnetism dissapears between 8 Å and 12 Å. Line shape and width, and g factor, were also measured.

E. E. HUBER, D. O. SMITH and J. B. GOODENOUGH: 'Domain-Wall Structure in 'Permalloy' Films'; pp. 294–5.

Domain-wall structure in 'Permalloy' films in the thickness range 25–2000 Å was studied by the Bitter technique.

Small Particles and Permanent Magnets

J. J. DE JONG, J. M. G. SMEETS and H. B. HAANSTRA: 'Some Results of an Electron Microscopical Study of the Metallographic Structure of Two Alloys for Permanent Magnets ('Ticonal G' and 'Ticonal X')'; pp. 297–8.

The microstructures of the two nickel-aluminium-cobalt-iron-base magnet alloys, in the condition giving optimum magnetic properties, were studied by the electron microscope, using the direct carbon replica. It has not yet been found possible to decide whether the structure in the optimum condition is a true two-phase type or a single-phase one with periodic fluctuations in composition.

K. J. KRONENBERG and R. K. TENZER: 'Relation between Colloid Pattern and Permanent-Magnet Precipitate during the Magnetization Reversal in 'Alnico V''; pp. 299–301.

Improved electron-microscope observation technique has confirmed some of the basic assumptions on the structure of 'Alnico V' and has added knowledge of details. The permanent-magnet precipitate was investigated at various states of its growth, starting from nucleation. Figures with details smaller than 20 Å reveal the completely developed structure of the alloy, which differs somewhat from descriptions which have previously been given in the literature. The observations made show clearly that the precipitated particles have, at that stage, grown together to a continuous anisotropic network subdividing the matrix. Structural effects occurring during reversal of magnetization are described.

R. K. TENZER and K. J. KRONENBERG: 'Phase Analysis of 'Alnico V' based on Temperature Effects'; pp. 302–3.

The authors attempt to provide a magnetic analysis of the two phases found in fully treated 'Alnico V.' (A composition of iron 52, cobalt 23, nickel 14, aluminium 8, copper 3, per cent., is taken as mean.) Structure-independent properties, e.g., saturation magnetization and temperature coefficient, are found to indicate the correctness of alloys supposed to represent these phases. A model of the two-phase structure of 'Alnico V' is derived from electron micrographs, and the combined saturation magnetization, and the combined temperature coefficients are calculated with this model, using the measured properties of the single alloys. The two alloys whose combined properties come closest to those of 'Alnico V' consist mainly of NiAl and Fe_2Co, respectively.

A. E. BERKOWITZ and P. J. FLANDERS: 'Magnetic Measurements on Some Precipitating Systems'; pp. 314–16.

In the systems Au/Ni and β-brass/Fe, ferromagnetic precipitate particles develop in a non-magnetic

matrix. Single-crystal samples of these alloys were examined by measuring saturation magnetization, torque curves, rotational hysteresis, and remanence. The data on the Au/Ni system were analysed to derive particle-anisotropy distributions, orientation of the 'easy' axes of the particles, and other features of the precipitate-particle system. Electron micrographs of the precipitate particles were found to be in general agreement with this analysis.

Ferromagnetic Resonance

J. R. WEERTMAN and G. T. RADO: 'Low-Temperature Spin-Wave Resonance at 3000 and 4000 Mc./sec. in a 'Permalloy' having Nearly Zero Magneto-crystalline Anisotropy'; pp. 328–9.

Earlier observations of spin-wave resonance in 'Permalloy' specimens of extremely low anisotropy were extended to liquid-nitrogen temperature. Measurements, at both room temperature and at the sub-zero temperature, were made at 3000 and at 4000 Mc./sec.

E. SCHLÖMANN and J. R. ZEENDER: 'Ferromagnetic Resonance in Polycrystalline Nickel-Ferrite Aluminate'; pp. 341–3.

Line broadening due to crystalline anisotropy and the random orientation of the crystallites was investigated for the case in which the anisotropy field is much larger than the saturation magnetization. Relevant theory is supported by observations made on nickel-ferrite aluminates.

Magnetic Alloys: Mostly Oriented

S. CHIKAZUMI: 'Magnetic Anisotropy Induced by Magnetic Annealing and by Cold Working of Ni_3Fe Crystal'; pp. 346–50.

After a brief survey of earlier work on magnetic annealing, a report is made of recent investigations on the magnetic anisotropy induced by cold rolling, using both single crystals and polycrystals of Ni_3Fe.

M. J. SAVITSKI: 'Effects of Composition and Processing Variables on the Magnetic Properties of the 50% Nickel-Iron Alloy'; pp. 353–5.

It is well known that manufacturers of oriented 50–50 nickel-iron alloys differ not only in their methods of melting and processing, but also in their preference for the minor elements which are to be incorporated in the alloys, and the amounts of such elements which are considered optimum. This paper reports a detailed study of the influence of some variables in composition and processing, as affecting the magnetic properties of this type of alloy, used widely in the cores of amplifiers.

The variables studied (in various combinations) were the following:

Composition: manganese 0, 0·5, or 1·0%
sulphur, 0 and 0·025% (added as FeS)
silicon, 0 and 0·25%
oxygen, 0 and 0·015% (added as Fe_2O_3).

Processing: (*a*) hot rolling at two levels of temperature (1050°–850°C. or 850°–700°C.);
(*b*) intermediate annealing for 2 hours at 1000°C. in argon, or not annealing;
(*c*) temperature of final annealing (six levels in the range 1000°–1250°C.).

The resulting ingots were processed to tape, from which toroidal cores were made. Tests on these cores indicate the beneficial influence of small additions of the minor elements: a binary manganese-oxygen combination gave the best results. Processing conditions must be adjusted to compositional variations: in general, hot rolling at a high temperature level was beneficial, and annealing after hot rolling was detrimental to magnetic performance.

P. K. KOH: 'Orientation Study of Ultra-Thin Molybdenum-'Permalloy' Tape'; pp. 356–8.

When ultra-thin molybdenum-'Permalloy' tape is used for switch-core and coincident-current memory devices, the squareness of the hysteresis loop and a minimum value of the switching coefficient depend on correct grain orientation. This paper contains a report of studies of the cold-deformation and annealed textures of such metal cores. Quantitative pole-density stereograms of (111), (220), (200) and (113) poles of 1/8-mil molybdenum-'Permalloy' tape were developed.

Magnetic Moments and Crystal Structures of Oxides

C. Q. ADAMS and C. M. DAVIS: 'Effect of Hydrostatic Pressure and Temperature on the Magnetic Properties of a Nickel-Zinc Ferrite'; pp. 372–3.

Magnetic properties of a nickel-zinc ferrite (15% NiO, 35% ZnO, 50% Fe_2O_3) were measured over a temperature range of 23°C.–110°C., at hydrostatic pressures up to 40,000 p.s.i. (18 t.s.i.; 28 kg./mm.^{2}).

J. B. GOODENOUGH, D. G. WICKHAM, W. J. CROFT: 'Some Ferrimagnetic Properties of the System $Li_xNi_{1-x}O$'; pp. 382–3.

A. J. WOLD, R. J. ARNOTT and J. B. GOODENOUGH: 'Some Magnetic and Crystallographic Properties of the System $LaMn_{1-x}Ni_xO_3+\lambda$'; pp. 387–9.

Applications and Testing

W. H. ROBERTS: 'Performance of Permanent Magnets at Elevated Temperatures'; pp. 405–7.

The effects of temperatures up to 600°C. and of vibration were studied on magnets of the 'Alnico V' and 'Alnico VI' types. The results indicate that both alloys may be used in most instrument and control applications involving temperatures up to 500°C., but that considerable loss in magnetic

properties would be expected at higher temperatures. The influence of sub-zero temperature (−70°C.) was also studied.

L. I. MENDELSOHN: 'Micro-Uniformity of Permanent Magnets'; pp. 407–8.

Description of a method of measuring variations in flux density normal to the pole faces of short axially-magnetized cylindrical permanent-magnets in materials of various types, including 'Alnico', 'Cunife' and 'Cunico' types. Directional materials such as cast 'Alnico V' show variations as high as 17 per cent.

Ferromagnetic Resonance: Non-Linear Effects and Garnets

This section contains no papers relating to nickel-containing materials.

Anisotropy and Magnetostriction

W. J. CARR: 'Temperature Dependence of Ferromagnetic Anisotropy'; pp. 436–7.

Discussion of the temperature-dependence of the crystalline anisotropy constants of iron, cobalt and nickel.

P. K. BALTZER and J. G. WHITE: 'Crystallographic and Magnetic Studies of the System $(NiFe_2O_4)_{1-x}+(NiMn_2O_4)_x$'; pp. 445–7.

The system $(NiFe_2O_4)_{1-x}+(NiMn_2O_4)_x$ was selected to study the rôle of the Mn^{3+} ion in determining the crystallographic and magnetic properties of spinels. The ionic distribution was investigated by means of X-ray diffraction.

C. J. KRIESSMAN, S. E. HARRISON and H. S. BELSON: 'Relationship between Single Crystal and Effective Polycrystalline Anisotropy Constants in Ferrites'; pp. 452–3.

The three materials studied in this work included a ferrite of the $NiFe_2O_4$ type.

Magnetization Processes: Reversals and Losses

F. J. SCHNETTLER and F. R. MONFORTE: 'Effect of Cobalt on the Relaxation Frequency of Nickel-Zinc Ferrite'; pp. 477–8.

Determination of the effect of cobalt on relaxation frequency and initial permeability of compositions varying in nickel, cobalt and zinc content, with constant iron.

T. D. ROSSING and V. J. KORKOWSKI: 'Switching Properties of 'Permalloy' Cores of Varying Coercivity'; pp. 479–80.

In the course of research designed to develop a 4-79 molybdenum-'Permalloy' core for use in a memory device with non-destructive read-out, studies were made of switching properties of 'Permalloy'-tape cores with coercivities ranging from 0·1 to 1·0 oersted, corresponding to heat-treatments over a temperature range of 300°–900°C. The switching time of a core can be shortened by means of auxiliary field pulses which help to nucleate domains of reverse magnetization, but have little effect on their rate of propagation. In general, auxiliary field pulses applied perpendicular to the direction of remanent magnetization are more effective than those applied anti-parallel to it, provided the latter are not large enough to cause switching by themselves. The timing of the auxiliary pulses is important.

F. G. WEST: 'Temperature Dependence of the Absorption of Ultrasound in a Nickel Single Crystal from 77° to 650°K.'; pp. 480–2.

The results are stated to show the inadequacy of current theories of absorption which are based on micro-eddy current losses accompanying domain-wall motion and domain rotation.

J. J. BROPHY: 'Magnetic Fluctuations in Molybdenum-'Permalloy' '; pp. 483–4.

Considerations of generalized noise and magnetic viscosity suggest the existence of magnetic fluctuations in ferromagnets. Magnetic fluctuations in molybdenum-'Permalloy' tape ring cores have been detected by observing noise voltages at the terminals of a coil wound on the sample. Observations on both thin- and thick-tape cores are reported.

Magnetic Apparatus and Techniques

A. H. BOBECK: 'The 'Twistor.' New Concept in Large-Size Memory Arrays'; pp. 485–6.

Three methods have been developed for storing information in a coincident-current manner on magnetic wire. The resulting memory cells have been collectively named the 'twistor'. Two of these methods utilize the strain-sensitivity of magnetic materials and are based on the well-known Wertheim or Wiedemann effects; the third utilizes the favourable geometry of a wire. The paper includes reference to experiments in which nickel wire was used as the strain-sensitive material.

D. H. WENNY and K. M. OLSEN: 'Casting of 'Alnico VII' with Improved Physical Properties'; pp. 504–5.

The alloy designated 'Alnico VII' has the following nominal percentage composition: aluminium 8, titanium 5, copper 3, cobalt 24, nickel 18, iron 42. This material has a residual induction, B_r, of 7000 gausses and a coercive force, H_c, of 1000 oersteds. This latter value is the highest attainable in any of this class of alloy and could be advantageously used in magnet structures, but, due to the inherent excessive brittleness of the alloy, full advantage has not been taken of these unusual properties.

The work now reported shows that it is possible, by controlling the initial freezing and cooling rates of the castings, to produce an alloy of this type in a condition completely free from cracks, and having sufficient cohesion to withstand the handling and thermal shock involved in heat-treatment. The desired retardation in cooling can be accomplished by the use of silica shell moulds pre-heated to temperatures above 1000°C. Good surface finish and close dimensional control are also secured by such procedure. The economics of the process, with regard to large-scale use, have still to be studied.

Magnetic Moments and Crystal Structures of Metals

J. S. KOUVEL, C. D. GRAHAM and J. J. BECKER: 'Unusual Magnetic Behaviour of Disordered Ni_3Mn'; pp. 518-19.

Measurements have been made of the magnetization of disordered Ni_3Mn for various fields between 945 and 7550 oersteds and at many temperatures between 1·5 and 300°K. (−271·5 and +27°C.). The magnetization-*vs*-temperature curve for any given field is found to have a pronounced maximum at about 25°K. (−248°C.) and a long concave-upward section extending from about 140°K. (−133°C.) to temperatures above 300°K. (+27°C.). By means of a new method of analysis, the Curie point is deduced to be 132°K. (−141°C.). An abnormally large induced magnetization, greatest at about 50°K. (−223°C.), is also found. All the experimental results are consistent with the occurrence of a ferrimagnetic-ferromagnetic transition at low temperatures.

Domain Pattern and Theory

This section contains no papers relating to nickel-containing materials.

Effect of Elevated Temperatures on Stability of Nickel-containing Permanent-Magnet Alloys

A. G. CLEGG and M. McCAIG: 'The High-Temperature Stability of Permanent Magnets of the Iron-Nickel-Aluminium System.'

Brit. Jnl. Applied Physics, 1958, vol. 9, May, pp. 194-9.

An earlier paper by the senior author had described the effect of sub-zero temperatures on the stability of permanent-magnet alloys of the 'Alcomax', 'Alnico', and cobalt-steel types (*ibid.*, 1955, vol. 6, pp. 120-3; see abstract in *Nickel Bulletin*, 1955, vol. 28, No. 6-7, p. 109). This later paper reports examination of the effects of temperatures up to 550°C., on the stability of five types of magnet alloy: see table below. For some of the alloys the influence of the ratio of length to cross section was also studied.

Using a differential ballistic method of examination (apparatus described and illustrated), the open-circuit magnetization of rectangular bars of these alloys was measured at various temperatures.

From the experimental results reported the authors conclude that heating magnet materials of these types up to temperatures not exceeding 550°C. causes no permanent metallurgical changes, but results in loss of magnetization. Such losses are partly irreversible, in the sense that they can be restored only by re-magnetization, and partly reversible, i.e., they are restored by cooling. Pre-ageing by an alternating field does not eliminate the irreversible losses.

On heating to any temperature up to 550°C., a magnet quickly reaches stability at that temperature, and suffers no further irreversible changes between room temperature and that temperature. The irreversible changes arise largely from changes in the demagnetization curve, and vary with dimension

Table I

Composition, Treatment and Properties of Magnets

Alloy	Average Composition (weight %; balance Fe) and Heat-Treatment	B_r(G)	(BH) max. (M.G.O.)	−B/H at (BH) max.	H_c (oersteds)
'Alcomax II'..	Al 8·1, Ni 11·9, Co 21, Cu 4·4	12700	4·7	23·0	565
'Alcomax III'	Al 7·8, Ni 13·2, Co 25, Cu 3, Nb 0·8	11900	5·14	18·1	662
'Alcomax IV'	Al 7·4, Ni 13·3, Co 24·5, Cu 3, Nb 2·5	11200	4·3	17·3	725
	Cooled from 1250°C. at 1·2 C.°/sec., in a magnetic field. Tempered 48 hours at 590°C., 48 hours at 560°C.				
'Alnico' ..	Al 9·7, Ni 17·5, Co 13·6, Cu 5·9	7500	1·68	12·7	605
	Air-cooled from 1250°C., tempered 2 hours at 600°C.				
'Alni'.. ..	Al 13·1, Ni 25·9, Cu 2	6060	1·21	10·0	520
	Air-blast cooled from 1250°C. Tempered 1 hour at 700°C.				

ratios and material, being appreciably less for the anisotropic alloys.

The reversible losses are approximately linear up to 200°C., with increasing curvature at higher temperatures. Temperature coefficients for all materials tested are of similar order, but tend to be less for magnets having short dimension ratios: possible reasons for this variation are suggested.

It was observed that the self-demagnetizing factor of a rectangular bar magnet is not constant, but changes with the induction at the central section.

Oxidation Characteristics of Iron-Nickel Alloys

M. J. BRABERS and C. E. BIRCHENALL: 'High-Temperature Oxidation of Iron-Nickel Alloys.' *Corrosion*, 1958, vol. 14, Apr., pp. 179t–82t.

The authors' previous experiments, supplemented by reference to the literature, had established that nickel-iron alloys of low nickel content oxidized to produce a layer of wüstite free from dissolved nickel and overlaid with layers of nickel-free magnetite and hematite. The nickel was enriched in the metal layer near the external scale and as the nickel content rose the subscale formed in this region, which was initially wüstite, became a spinel. When the supply of oxygen was withheld and equilibrium conditions were attained by continued heating, the oxide was entirely converted to wüstite except for small amounts of spinel which lined cavities in the subscale region.

In the earlier work (see *Jnl. de Chimie Physique*, 1956, vol. 53, pp. 810–16; *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 47), a fairly thick oxide layer was obtained by oxidation of the alloy in air or oxygen for three days, and, after flushing with argon, the system was sealed off under the inert atmosphere and equilibrated for a week to ten days. The series of experiments reported in this later paper were conducted in an attempt to confirm the validity of, and to supplement, the results already obtained. The basic procedure used was the same as that employed in earlier experiments, but a closer degree of control was maintained. Oxidation was effected at 1050°C., in very brief stages, with intermediate partial equilibration periods, and the final equilibration period was extended to more than 1000 hours.

The subscale was found to form preferentially at grain boundaries, and equilibration occurred mainly by dissolution and diffusion of oxygen in the alloy phase. Porosity developed in the subscale region during equilibration, but sintered out as a result of longer heating.

On pure iron, at temperatures above 700°C. wüstite grows twenty times faster than magnetite, and it is concluded that since, in the case of the alloys, the rate of growth of wüstite largely determines the overall rate of growth of the composite oxide scale, the lowering of the wüstite content achieved by the presence of nickel in the metal phase will result in a proportionate decrease in the rate of oxidation, which will eventually be slowed down still further by nickel enrichment just below the scale. If the rate of growth of the nickel-containing spinel is similar to that of magnetite, elimination of wüstite will mean a twenty-fold reduction in the oxidation rate.

The iron-rich corner of the ternary phase diagram derived from the results of the experiments at 1050°C. is presented and discussed, and, as a result of the observations made, the authors propose a more detailed mechanism for the oxidation of iron-nickel alloys and for the homogenization of the resulting composite.

CONSTRUCTIONAL STEELS

Production of High-Grade Iron Ore at International Nickel Company's Plant

See abstract on p. 183.

Hydrogen Embrittlement of Steel

J. G. MORLET, H. H. JOHNSON and A. R. TROIANO: 'A New Concept of Hydrogen Embrittlement in Steel.'

Jnl. Iron and Steel Inst., 1958, vol. 189, May, pp. 37–44.

This paper is identical with that published by the same authors as *Wright Air Development Center Technical Report* 57-190, Mar. 1957, an abstract of which appeared in *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 49.

Low-Alloy Nickel-containing Steel for Welded Bridge Construction

L. C. HOLLISTER: 'Design and Fabrication by Welding of the Carquinez Strait Bridge.'

Welding Jnl., 1958, vol. 37, Apr., pp. 309–19.

The new bridge now being built by the California Division of Highways across Carquinez Strait is believed to be the first major cantilever truss-type structure in which the members are fabricated by welding. The bridge consists of two main spans each 1,100 ft. (330 m.) long, with two anchor arms 500 ft. (150 m.) long, and a central tower span of 150 ft. (45 m.), making a total length of the main spans across the strait 3,350 ft. (1,005 m.). Dimensional details and design of the bridge are described and illustrated in this article.

The new structure represents the latest ideas in modern design, making use of ultra-high-strength steels for the spans, high-tensile steel for bolts, and welded fabrication of the truss members. The weldable high-strength steel which has been selected for use is the U.S.S. 'T-1' type, a low-alloy type containing carefully controlled amounts of nickel, chromium, molybdenum, vanadium and copper. The steel has a minimum yield strength of 90,000 p.s.i. (40 t.s.i., 63 kg./mm.^{2}). Substantial economies have been effected by the use of this material, combined with welded construction.

Nickel-base Alloys for Aeronautical Applications

See abstract on p. 194.

HEAT- AND CORROSION-RESISTING MATERIALS

Nickel-base Alloys for Aeronautical Applications

T. E. KIHLGREN: 'A Review of Nickel-base Alloys for Aeronautical Applications.'

Reprint, by NAT. STANDARDS ASSOCN., from *Report of High-Strength, High-Temperature Materials for Standard Parts Symposium;* 45 pp. including discussion. Issued by INTERNATIONAL NICKEL CO., INC.

The background against which this paper was written is set by:

A graph indicating the increased operational temperatures which, by the end of the present decade, may be expected in various components of a gas-turbine engine.

A diagram showing components of gas turbines for which nickel-base alloys are currently used or are potentially useful.

Graph and diagram are related, in that rise in temperature, and thus increased efficiency, have been achieved only by development of alloys capable of withstanding the more severe conditions involved. The suitability of nickel-base alloys for gas-turbine components is well known. Increase in the speed of aircraft will in future involve a correspondingly greater degree of aerodynamic heating: airframe temperatures will eventually approach 1200°F. (650°C.), and at these temperatures the nickel-base alloys will become of significance also as possible replacements for precipitation-hardenable stainless steels now used in certain aircraft structural applications. The author's aim is therefore to make a general review of the high-temperature properties of the nickel-base alloys, to emphasize certain characteristics, and to indicate the forms in which they are of greatest interest for airframe parts.

High-temperature 'nickel-base alloys' are defined as those containing at least 40 per cent. of nickel, rendered age-hardenable by additions of aluminium and titanium. The information given relates mainly to the 'Nimonic', 'Inconel' and 'Incoloy' series. A considerable part of the paper consists of comprehensive graphical data on the mechanical and physical properties of alloys in cast and wrought forms, including a section on sheet materials.

The alloys are discussed first as a generic group, and the high-temperature characteristics are outlined by reference to the 100-hour rupture strengths of the respective alloys forming part of the precipitation-hardened nickel-base group.

Attention is then directed to the properties of individual alloys which hold promise as possible materials of construction for aircraft structures. Not all such alloys can be fabricated into sheet, but 'Inconel X' and 'Incoloy 901' are considered to have an important potential. The author's brief evaluation of such materials is supplemented by reference to rupture data derived from the alloys in sheet form.

Resistance of the high-temperature materials to static and cyclic oxidation conditions is surveyed, and mention is made also of the availability and applications of alloys suitable for investment casting, e.g., 'Inconel 713C'.

Finally, the influence of heat-treatment is discussed in relation to properties, with particular reference to 'Inconel X' and 'Inconel 100'. In particular, attention is drawn to the importance of the 'cross-over temperature', i.e., the critical temperature which separates the heat-treatment optimum for production of properties suitable for alloys operating at relatively low temperatures from that which gives optimum properties in the same alloy at higher temperatures.

The paper ends with a brief practical review of processing methods: forging and hot-working, heat-treatment, forming, machining, welding and brazing.

(Information in some sections of this paper is closely cognate with that given in the review by K. B. YOUNG, published in *Canadian Aeronautical Journal*, 1957, vol. 3, Oct., pp. 287–95; abstract in *Nickel Bulletin*, 1958, vol. 31, No. 5, p. 140.)

Oxidation of Nickel-Aluminium Alloys

E. I. MOZZHUKIN, L. K. PIVOVAROV and YA. S. UMANSKII: 'Oxidation of Alloys containing the Compound NiAl.'

Zhur. Priklad Khim., 1957, vol. 30, pp. 1593–9.

A study was made of the behaviour of nickel-aluminium and cobalt-aluminium alloys, some cast, others made by powder-metallurgy methods, and of nickel. Heat-resistance was evaluated on the basis of gain in weight, and the surface condition of the specimens was examined after exposure. Some observations made on the resistance of various alloys to deterioration under exposure at 1000°, 1100° and 1200°C. are recorded in *Chemical Abstracts*, 1958, vol. 52, pp. 4448–9.

Corrosion by Vanadium Pentoxide: Literature Review

K. SACHS: 'Accelerated High-Temperature Oxidation due to Vanadium Pentoxide.'

Metallurgia, 1958, vol. 57, Mar., pp. 123–37; Apr., pp. 167–73; May, pp. 224–31.

During the war period development of the gas turbine centred on its aircraft applications, i.e., on turbines operating on relatively pure fuels. Later the experience so gained was utilized in design of experimental industrial gas turbines, in which the use of comparatively cheap residual-oil fuels is the prime factor in making such units a commercial proposition in face of competition from the steam turbine. In 1948 reports began to be published relating to corrosion of blading resulting from the presence of vanadium pentoxide in the ash from the less refined fuels, and it became obvious that this factor would be a serious hindrance to advancement of the gas turbine in land and marine use. The

nature of the problem, and possible remedial methods, have since that time been the subject of intensive research.

This comprehensive review, based mainly on the literature, supplemented by the author's personal experience, summarizes the main features of the information which has been published. The survey is supported by a *bibliography* of 41 references: it is well illustrated, and includes much tabular, graphical and other data derived as a result of research on the behaviour of austenitic chromium-nickel steels and nickel- and iron-base alloys in conditions involving attack by vanadium pentoxide. The author makes ample quotation from original sources in order to present 'a reasonably balanced view of the present position'.

The first section of the review includes a table showing typical analyses of residual-oil ash stemming from oils available in various countries. The survey demonstrates the world-wide nature of the vanadium-pentoxide problem, but attention is directed to the large supplies of crude oil of relatively low vanadium content which are available to the U.S.A., thus making the difficulties less acute in that area.

A detailed account is given of the nature and extent of the deleterious effects which have resulted from the use of residual oil fuel in experimental gas turbines, and this review is followed by a description of procedure employed in laboratory studies made to supplement the practical experience. Variables which are generally acknowledged to be important in connexion with vanadium-pentoxide attack are composition of the alloy of which the component is made, time of exposure, temperature, total amount of corrodent present, presence of other reagents with the vanadium pentoxide, and the presence of sulphur-bearing gases. The effect of composition of material is illustrated by a discussion of the results of research by various investigators: comparative experiments have indicated that nickel-base alloys of the nickel-chromium-type are somewhat less 'catastrophically' attacked than iron-base materials.

Certain constituents which may be associated with vanadium pentoxide can have a markedly intensifying effect on corrosion: the most important of such media are sodium salts, sulphur-bearing gases or salts, and chlorides. Information on the influence of the respective constituents is reviewed.

Theories advanced to interpret the mechanism of the attack have been many and varied: an outline is given of evidence which has been presented in support of the most generally accepted view, which attributes the deterioration to dissolution of the protective oxide film on the alloys by molten V_2O_5, acting as a flux. Alternative mechanisms are critically considered.

From the conglomerate mass of data and theory now available, the author lists the following accepted facts: the presence of a liquid phase plays an important part in the corrosive attack; the protective effect of chromic-oxide films is eliminated; 'catastrophic oxidation' takes place at the metal/scale interface; the surface scale is spongy, porous and in an abnormally high state of oxidation.

Among the secondary effects of vanadium pentoxide reported in the literature and discussed in this review is the observation that accelerated oxidation can result not only from direct contact with V_2O_5, but also from its mere proximity. Evidence is reviewed which suggests that the corrosive tends to search out weak points on the surface of a metal, resulting in highly localized attack. The peculiarly 'antagonistic' reaction of vanadium pentoxide with steels containing molybdenum is also considered.

The review ends with a résumé of attempts which have been made, or proposals which have been put forward, to solve the problem of vanadium-pentoxide corrosion. While it is recognized that the obvious and most effective solution would be to ensure that the fuel oil was free from vanadium, such a method is not practicable. Other more feasible procedures suggested have included removal of the harmful ingredients from the ash, by some form of separator between the combustion chamber or flame tubes and the turbine. Efforts have been concentrated also on the search for additives which, by formation of high-melting-point compounds with vanadium pentoxide, would ensure that the harmful fluxing effect could operate only at temperatures above the practical service limits. The results of tests with various additives are summarized.

From the metallurgical point of view the solution has been sought by modification of composition of materials of construction, and in this connexion reference is made to development work on nickel-base alloys, and to evaluation of the degree of protection afforded by electrodeposited nickel and chromium coatings, and by chromized, aluminized or siliconized coatings.

In conclusion, the author urges the need for further systematic research, with the aim of developing an alloy which not only is resistant to attack by vanadium pentoxide, but also retains all the properties essential to its primary function as a material of construction for gas-turbine components.

Properties and Uses of Resistance-Heating Alloys

R. J. FABIAN: 'How to Select a Resistance-Heating Alloy.'

Materials in Design Engineering, 1958, vol. 47, Feb., pp. 104–8.

The article reviews the technical and economic advantages and the limitations which have to be balanced in determining the most suitable electrical-resistance alloy for a specific heating application. Four main classes are considered: (1) nickel-chromium; (2) nickel-chromium-iron; (3) iron-chromium-aluminium-(cobalt); and (4) molybdenum silicide. The section concerned with the nickel-chromium-iron system is sub-divided, on the basis of nickel and chromium content, into two grades, dealing (*a*) with alloys containing nickel 60–62, chromium 15–16, per cent., and (*b*) those containing nickel 35, chromium 18–20, per cent.

Notes are given on the properties of the material,

cost factors, and service conditions which might deleteriously affect the life or performance of the respective alloys. This information is supplemented by tabular and graphical data and by practical hints on the design of heating elements.

Nickel-Alloy Heat- and Corrosion-Resistant Electrodes and Filler Wires

The following specifications were issued on Apr. 15, 1958, by the SOCIETY OF AUTOMOTIVE ENGINEERS in their Aeronautical Materials series:

'Alloy Wire, Corrosion- and Heat-Resistant, Nickel-base 22 Cr - 1·5 Co - 9 Mo - 0·6 W - 18·5 Fe.' *A.M.S.* 5798

'Welding Electrodes, Coated, Alloy, Corrosion- and Heat-Resistant, Nickel-base 22 Cr - 1·5 Co - 9 Mo - 0·6 W - 18·5 Fe.' *A.M.S.* 5799

The electrodes are intended primarily for metallic-arc welding of heat- and corrosion-resisting steels and alloys of similar or dissimilar composition. The wire is for use as bare-wire filler material for inert-gas, metallic-arc and tungsten-arc welding of the same range of materials.

The compositional limits specified for the wire are: carbon 0·05–0·15, silicon 1·00 max., manganese 1·00 max., phosphorus 0·040 max., sulphur 0·030 max., chromium 20·50–23·00, cobalt 0·50–2·50, molybdenum 8·00–10·00, tungsten 0·20–1·00, iron 17·00–20·00, per cent., remainder nickel.

The electrodes, for which quality and other requirements, including coatings, are laid down, are to be capable of depositing weld metal within the limits of composition shown for the filler wire.

Resistance of Nickel-containing Materials to Corrosion by Molten Sodium/Potassium Mixture

S. J. BASHAM, J. H. STANG and E. M. SIMONS: 'Corrosion Screening of Component Materials for Na/K Heat-Exchange Systems.'

Nuclear Engineering and Science Conference, Chicago Mar. 1958, *Preprint* 24; 25 pp.

Mixtures of molten sodium and potassium are highly corrosive, a property which has hindered attempts to put to practical use the many characteristics which make them suitable as high-temperature heat-transfer media. Although research has shown that a relatively large number of metals and alloys (*inter alia*, those of nickel base) exhibit satisfactory resistance to attack at temperatures up to 1650°F. (900°C.), and are therefore suitable for normal plumbing applications, there has been little success in the search for materials which will be relatively immune to seizing, galling, or self-welding in such critical applications as valve-seat inserts, valve plugs, shaft-seal facings and bearings.

This report presents the results of screening tests to determine the resistance of 61 materials to corrosion by Na/K, with a view to their further evaluation as possible materials of construction for valves and other such components in high-temperature flow systems.

The materials tested were of four general types:

(1) High-temperature alloys, comprising 'Hastelloy B', 'Inconel X', 'Kanthal', 'Stellite 6' and 'Stellite 93', and 'Thermenol' I, II and III;

(2) Refractory and noble metals (molybdenum, niobium, rhenium, tantalum, vanadium, tungsten and zirconium);

(3) Cermets consisting of carbides of tungsten, titanium or molybdenum as the refractory, and nickel or cobalt as the metallic binders;

(4) Ceramics (mainly metallic carbides, borides and oxides).

Specimens were tested in the hot end of a sealed capsule (fabricated from fully heat-treated 'Inconel X') which was partially filled with Na/K. The capsule was placed in a tilting furnace and rocked, to cause the liquid metal to flow from the hot end to the cool end and back, thus simulating a thermal cycle. The temperatures in the hot and the cool ends were, respectively, 1600°F. (870°C.) and 1200°F. (650°C.).

The results are presented in tables in which each material is graded, on the basis of corrosion-resistance, in one of three classes, according to its potential usefulness for components in the service envisaged.

The refractory metals, particularly molybdenum and tungsten, showed good resistance to attack, and, in general, those tested are considered by the authors to show excellent promise. All the alloys except 'Stellite 93' were unaffected by the corrosive, but, on the basis of past experience, it is considered unlikely that they would show the necessary resistance to galling, self-bonding and wear. In the cermet groups the carbides showed the best corrosion-resistance: tungsten carbide was particularly promising. To obtain still better corrosion-resisting quality it is desirable to lessen the porosity of cermet materials. Of the ceramics tested, only a few carbides showed evidence of satisfactory resistance to corrosion.

Precipitation-Hardenable Stainless-Steel Corrugated Foil for Aircraft

'Paper-Thin Steel Sheet Cuts Weight of Aircraft.' *Materials in Design Engineering*, 1958, vol. 47, Apr., pp. 128–33.

The article discusses the production, design and applications of corrugated stainless-steel foil developed by the Ryan Aeronautical Company as a material of construction for aircraft structures. The information it contains is fundamentally similar to, though more detailed than, that given in an article by B. MITCHELL, in *Iron Age*, 1958, vol. 181, Feb. 6, pp. 102–4 (see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 6, p. 172).

Comparison of 18-8 Chromium-Nickel and Low-Nickel High-Manganese Stainless Steels

G. A. SANDS and M. B. KEADY: 'How the '200 Series' Compares with 18-8 Stainless Steel.'
Materials in Design Engineering, 1958, vol. 47, Apr., pp. 120-3.

Since 1955, when the chromium-nickel-manganese stainless steels were first recognized by the American Iron and Steel Institute as a new standard class of steels (the '200 series') production has considerably increased.

This article compares the A.I.S.I. 200 and 300 (Cr-Ni) series on the basis of mechanical properties, corrosion-resistance, fabricating characteristics, (formability, weldability and finishing characteristics), and cost. Tabular data on the relative resistance of the two series to corrosion by various media are also included.

Influence of Irradiation on the Properties of Metallic Materials

C. A. BRUCH and W. E. McHUGH: 'Radiation-Damage Studies of Seven Non-Fissionable Metals.'
Nuclear Engineering and Science Conference, Chicago, Mar. 1958, *Preprint* 37; 69 pp.

Research on radiation damage in metals has been in progress for some years at the Knolls Atomic Power Laboratory. The metals selected for study were chosen as representative of the three major crystallographic systems (copper, nickel, titanium, zirconium, iron and molybdenum), and a typical molybdenum - containing chromium - nickel stainless steel was also included. Tensile specimens of each material have been irradiated, under varying conditions of exposure, in three types of nuclear reactor, and measurements have been made of resultant changes in hardness, electrical resistance, tensile properties and microstructure. An interim report, giving some tentative results, was published in 1956; see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 1-2, p. 1.

The purpose of the present paper is to summarize the results of test measurements made to date and to discuss conclusions which have been drawn from the data obtained. Full details are given of the irradiation procedures, the test conditions, and the properties of the materials before and after irradiation. In addition to the data on the individual metals, a comparison is made of the high-energy neutron-flux values obtained by the two different methods of irradiation employed.

The general effects of irradiation were to increase the hardness, strength and electrical resistance of the materials tested, and to lower their ductility. In many cases (including copper, nickel, iron and stainless steel) a drop-in-load type of yield point was produced. Load-elongation curves were raised to higher levels, and the amount of uniform elongation was much reduced, or, in some cases, eliminated. In several materials (e.g., iron, molybdenum and stainless steel) it was found that under the influence of irradiation electrical resistance first increased but subsequently, with increasing irradiation, decreased.

From the results it is concluded that, other than radioactivity, no unique properties are produced in the materials by irradiation, and that equivalent properties can be obtained by the conventional techniques of alloying and/or cold working.

The authors propose a mechanism to interpret the qualitative effects of irradiation and discuss the influence of various irradiation parameters on the magnitude of radiation damage. In considering the data with a view to evolving some empirical relationship common to all the materials studied, reference is made to the observation that the maximum percentage increase in yield strength was inversely related to the original yield-strength value and that percentage change in electrical resistance was apparently dependent on the crystal structure of the metal. (It is noted that 'A' nickel exhibited an unusually large increase in yield as a result of irradiation.)

Influence of Uranyl-Sulphate Corrosion on Fatigue Strength of Stainless Steel

C. H. GABBARD: 'Diaphragm Feed Pumps for Homogeneous Reactors.'
Nuclear Engineering and Science Conference, Chicago, Mar. 1958, *Preprint* 74; 15 pp.

This paper discusses problems encountered in development of a 1·5 g.p.m. high-pressure, low-capacity diaphragm feed pump at the Oak Ridge National Laboratory. The main difficulties met pertained to design of suitable check valves for water and uranyl sulphate, and to means for ensuring long-term trouble-free service. The author discusses (1) contour shape and stress present in the diaphragm, (2) a method for studying the dynamic deflection of the diaphragm, and (3) the effect of corrosion by uranyl-sulphate solution, on the endurance limit of niobium-stabilized 18-8-type stainless steel (A.I.S.I. 347).

In the study of corrosion effects tests were made on the Type 347 steel in sheet form, using annealed and $\frac{1}{4}$, $\frac{1}{2}$, $\frac{3}{4}$ and full-hard material. The results indicate that the endurance limit is appreciably lowered by contact with the uranyl-sulphate solution. Further work is in hand on the same steel in the full-hard condition (which showed the best behaviour in the screening tests) and on the precipitation-hardening steel 'A.M. 350,' which, in air, has an endurance limit much higher than that of the steel already tested.

Resistance of Nickel-containing Materials to Corrosion by Uranium Hexafluoride

D. HEYMANN and F. E. T. KELLING: 'Corrosion of Some Metals and Alloys in Uranium Hexafluoride.'
Corrosion Technology, 1958, vol. 5, May, pp. 148-51, 158.

The experiments described in this paper were carried out to provide quantitative data which would supple-

ment the relatively large amount of qualitative information available on the resistance of various materials to corrosion by uranium hexafluoride. The following materials were tested: aluminium alloys; carbon, low-alloy and stainless steels; nickel; copper, brass, 'Monel' and the nickel-aluminium bronzes 'Hidurax 1' and 'Hidurax 4'; titanium-base alloys.

The specimens were exposed in a conventional uranium-hexafluoride-distillation apparatus at 80°C., a temperature at which the fluoride is liquefied and its vapour pressure is about 2·4 atm. The gas phase was not agitated.

Deposition of uranium tetrafluoride from the gas made it impossible to determine, merely by weighing the specimens before and after corrosion, the degree to which metal fluoride had formed. The procedure ultimately adopted comprised washing the surfaces clean of uranium compounds, dissolving the metal fluoride, and measuring the weight loss relative to that of uncorroded specimens subjected to the same treatment. Details are given of the procedures used for the respective types of material.

The results are presented in the form of tables showing depth of surface penetration as a function of time; the periods of exposure varied up to two months. The aluminium- and the titanium-base alloys exhibited good resistance to attack. Resistance of the carbon steels was also satisfactory and that of the 18-8 chromium-nickel steels was excellent. The results appear to indicate that in the carbon steels a high carbon content has a slight deleterious effect, and that the presence of chromium substantially diminishes resistance to corrosion. Addition of nickel, on the other hand, has a pronouncedly beneficial influence. Nickel, copper, brass and 'Monel' gave extremely low rates of corrosion, and whereas the copper and brass specimens were discoloured after exposure, the surfaces of the 'Monel' and nickel samples were unaffected.

Subsidiary experiments were carried out to investigate the growth of crystals on the specimens during exposure, and the superficial changes occurring after exposure at 80°C. or at room temperature. In this connexion the authors describe an optical apparatus by which it was possible to determine such effects, and to demonstrate the slight changes occurring on the surfaces of aluminium, 'Monel', nickel and stainless steel compared with the considerable alterations produced on the surfaces of carbon steels.

Effect of Plastic Deformation on Austenite→ Martensite Transformation in High-Alloy Steels

G. V. KURDYUMOV, O. P. MAKSIMOVA and N. I. NIKONOROVA: 'The Activating Influence of Plastic Deformation upon the Martensite Transformation.' *Doklady Akad. Nauk S.S.S.R.*, 1957, vol. 114, pp. 768-71.

The work described consisted in study of the influence of various degrees of deformation on transformational effects in two steels, containing, respectively, 18 per cent. chromium with 8 per cent. nickel and 17 per cent. chromium with 9 per cent. nickel. Evaluation was made by magnetometric methods. The results are summarized in *Chemical Abstracts*, 1958, vol. 52, p. 2702.

Nickel Alloys for Handling Fluorine and its Compounds

'Handling Fluorine and Fluorine Compounds with Inco Nickel Alloys.'

Published by INTERNATIONAL NICKEL CO. INC., May 1958; 24 pp.

Fluorine and its compounds are assuming an increasing importance in a wide range of applications in the chemical, petroleum and atomic energy industries. In view of the extremely corrosive nature of the substances handled, their satisfactory use is contingent on judicious selection of the materials of construction with which they come into contact. This publication summarizes corrosion data and industrial experience collated from a variety of sources, with the intention of indicating the usefulness of nickel-base alloys in this field. The information is given in four sections, the scope of which is noted below.

I. *Fluorine—Why it Presents Severe Corrosion Problems*

This introductory section briefly reviews the events which led up to the isolation of fluorine, comments on some of the properties of the element, and discusses the resistance of fluorocarbons to corrosion by fluorine itself.

II. *Fluorine—Generation, Use and Disposal*

After a short discussion of the method employed to passivate structural materials used in the manufacture of fluorine, attention is directed to the suitability of nickel and 'Monel' for anodes and containers in electrolytic cells used for fluorine generation; for pumps, piping, cylinders and valves employed to handle fluorine; and for plant connected with the disposal of the element by reaction with superheated steam. The section includes data on the corrosion-resistance of various materials exposed to fluorine at elevated temperatures.

III. *Hydrofluoric Acid—Wet and Dry*

The corrosive behaviour of anhydrous hydrofluoric acid is distinguished from that exhibited by dilute aqueous solutions of the acid, and reference is made to data derived from laboratory corrosion tests on the resistance of nickel-containing alloys and steels. In connexion with a description of the rôle of the acid in alkylation processes used in petroleum refineries, these data are later supplemented by details of comprehensive tests conducted under operating conditions in critical sections of three alkylation plants. Consideration is given to the specific resistance, to hydrofluoric acid, of the following materials: 'Monel'; 'K', 'H' and 'S' grades of 'Monel'; nickel; and 'Inconel'. Finally, the stress-corrosion cracking of high-nickel alloys and its prevention are discussed. Corrosion data are

tabulated to demonstrate the resistance of various metals and alloys to anhydrous hydrogen fluoride at elevated temperatures.

IV. *Future of Fluorine Chemistry*

This final section is concerned with the rôle of nickel-base alloys with respect to the atomic energy applications of fluorine and its compounds, i.e., handling of fluorine and molten fluorides, processing of uranium ore and atomic fuel, and preparation of boron-10. Notes are also given on methods of svnthesizing fluorocarbons.

Complex Chromium-Nickel Stainless Steels in Sulphuric-Acid Plant

A. A. BABAKOV and E. V. ZOTOVA: 'Corrosion of Chrome - Nickel - Molybdenum - Copper Steel in Production of Low-Nitroso-Sulphuric Acid.'
Khim. Prom., 1957, pp. 238–40.

Examination of the behaviour of heat-exchanger coils employed in cooling sulphuric acid (0·05 per cent. HNO_3) led to recommendation of steel containing chromium 19–23, nickel 23–28, per cent. as giving best resistance to attack and being suitable for cooling plant in which the wall temperature does not exceed 80°C. The steel should be used in the condition as quenched from 1150°C.

Chromium-Nickel Stainless Steels in Coal-Chemical Plants

C. P. LARRABEE and W. L. MATHAY:
'Controlling Corrosion in Coal-Chemical Plants.'
Corrosion, 1958, vol. 14, Apr., pp. 183t–6t.

The authors review the methods used to control corrosion in the coal-chemical plants of United States Steel Corporation: in particular, attention is directed to corrosion-resisting materials most suitable for use in structures and plant exposed to attack (*a*) by the atmosphere, (*b*) by coal, and (*c*) by chemicals encountered in such plant. The sections relating to behaviour in contact with the three types of corrodent are sub-divided under the heads of the plant concerned.

Consideration is first given to the protection of structural steelwork exposed to atmospheric corrosion, and in this connexion the most economical and effective methods of utilizing paint coatings are discussed. Reference is made also to the use, for outside structures, of copper-containing steels and other high-strength, low-alloy steels: galvanized steel and stainless steels for roofing and panelling are also considered.

Resistance to corrosion by moist coal, a prerequisite of any material of construction used for coal bunkers, chutes and hoppers, is governed mainly by ability to resist attack by ferric sulphate, which is the principal corrosive constituent of coal. Steels containing about 12 per cent. or more of chromium are considered to be ideal for such applications.

Plant in which severe corrosion due to contact with chemicals is likely to occur includes primary coolers, ammonia saturators, light-oil-recovery equipment, tar stills and pyridine-rectifier/tar plant.

In the primary coolers gases and vapours are cooled from 200° to 100°F. (95° to 40°C.), either by direct contact with 'ammonia liquor' or with metal tubes through which cooling water is passed. Corrosion of the exterior surfaces of such tubes is usually negligible, but in one coal-chemical plant severe attack occurred on the interior surface of tubes in the hottest portion (temperature 150°F.: 65°C.). It was found that the corrosion resulted from the use of river water contaminated with acid coal-mine drainage, to which lime had been added to raise the pH to about 4·5. Service and laboratory tests made to evaluate the resistance of galvanized, copper-lined and 18-8 chromium-nickel steels to such a solution showed that the life of the stainless steel would be practically unlimited. Since this latter steel, due to its scaling-resistance, also has better heat-transfer characteristics than galvanized steel, it has now been adopted for service in that plant.

A low-carbon steel containing chromium 17, nickel 12, molybdenum 2·5 per cent. has proved ideal for use in the ammonia saturators, in which the coke-oven gas leaving the primary coolers is bubbled through an aqueous solution saturated with ammonium sulphate and containing about 4–8 per cent. of free sulphuric acid. Tar stills, in which the tar is fractionated into carbolic oils, creosote and pitch, were previously made in cast iron, but, following corrosion tests, new tar stills have been constructed from the low-carbon stainless steel. The tar fraction contains inorganic chlorides, and temperatures are of the order of 480°F. (250°C.): a stress-relieving treatment is therefore necessary, to obviate stress corrosion. In the pyridine-recitifier/tar plant, in which pyridine bases are recovered from tar, lead-lined steel has been found to be considerably more resistant than stainless steels.

In 'light-oil' scrubbers the 'light-oil' (containing benzol, toluol and xylol) is adsorbed from coke-oven gas by direct contact with a petroleum or creosote 'wash oil'. Use of the creosote 'wash oil' resulted in severe corrosion, and in this connexion the authors discuss results of corrosion tests on carbon, chromium, and chromium-nickel steels exposed to this medium. The chromium-nickel steels were relatively unaffected, and carbon-steel equipment previously used is being replaced by parts made from straight chromium-nickel and molybdenum-containing types. Changes in process conditions, such as prevention of contamination by water and ammonia from the saturators, have also helped to minimize corrosion.

Stainless-Steel Pumps in Copper-Plating Plant

E. A. SCHOEFER: 'Cast Stainless Pump Handles Corrosive Electrolytes.'
Plating, 1958, vol. 45, Apr., pp. 366–7.

Continuous heavy-copper plating of steel wire,

in the plant of Western Electric Company, Baltimore, gave rise to serious problems, on account of the corrosive nature of the electrolyte, which comprises a solution of copper fluoborate containing approximately 5 oz./gal. of fluoboric acid and 20 oz./gal. of copper. The solution has a pH of less than 1, and is used in the range 120°–135°F. (50°–56°C.). The materials found most suitable for the pumps are cast stainless steels of the 29-20-2-3 nickel-chromium-molybdenum-copper and 20-11-2·5 chromium-nickel-molybdenum grades (Alloy Casting Institute designations CN-7M and CF-8M).

Extensive use has also been made of the same steels in components of other parts of the plating plant, e.g., in the cleaning and pickling sections, and in the finishing operation, when the wire is passed through a hydrofluosilicic-acid bath, to remove oxide formed during the annealing applied after production plating. A further use of the CN-7M-type steel is in handling the lead-fluosilicate solution from which the copper-coated wire is subsequently lead-plated.

See also

Stainless Steel in Pickling Equipment

'Cast Stainless Steel Stands up to Pickle Liquor.' *Steel*, 1958, vol. 142, Apr. 14, p. 141.

This article also refers to use of the cast alloy CN-7M (see above).

In pickling of stainless-steel wire, solutions used include nitric/hydrofluoric-acid mixtures, nitric-acid, sulphuric-acid and special de-scaling salt solutions. Many of these are used at elevated temperatures. To support the wire during the processing, cast hooks of CN-7M have been found specially suitable.

In production of silicon steel for electrical appliances, strip is fed as an endless ribbon through degreasing, rinsing, acid-pickling and final-rinsing tanks, to remove mill scale and oxides. Here the same cast corrosion-resisting steel proved to be the most suitable material for pumps, from the storage stage through to disposal of the spent acid.

In the stainless-steel pickling cycle the concentrated acid flows by gravity from the storage tanks to the pickling tanks, in which it is mixed to form a solution containing about 1% hydrofluoric acid and 3–4% nitric acid, which is used at 150°C. The spent mixture from the pickling tanks is pumped to a common collecting tank which also receives spent acid which has been used in pickling of the silicon steel, and this spent-acid mixture is pumped to another tank, in which it is neutralized by milk-of-lime. Here the CN-7M alloy is again used, for the pumps and valves which re-circulate the acid and milk-of-lime mixture in the neutralizing tank.

With regard to the economies effected by the use of corrosion-resisting alloys, it is estimated that the installation of the CN-7M pumps has, in 12 years' service, reduced maintenance cost to a degree equal to the cost of two new pumps.

Tungsten-Arc Welding of Ultra-Thin Precipitation-Hardening Stainless Steel

J. C. COLLINS and S. P. JENKINS: 'Tungsten-Arc Welding of 0·002-in. and 0·005-in. Stainless Steel and Titanium.'

Welding Jnl., 1958, vol. 37, Apr., pp. 324–7.

The article gives a detailed and well illustrated report on the technique developed for welding ultra-thin-gauge '17-7 P.H.' stainless-steel sheets used in making the exterior skins for the brazed honeycomb panels in the Convair B-58 supersonic bomber aircraft. To satisfy aerodynamic requirements, the weld must be made without filler material and, in addition, the weld-bead reinforcement must be held to a tolerance of −0·001 or +0·003 in. (−0·025 or +0·075 mm.) for the 72-in. (180-cm.) lengths required. The type of fixturing and the slow-speed, low-current operating technique adopted ensure maximum control of variables, and keep distortion to a minimum. Typical examples of the welded construction are shown.

Fatigue Tests on Brazed Joints of Stainless Steel

R. G. ASPDEN and W. FEDUSKA: 'Fatigue Characteristics of Single-Lap Joints of A.I.S.I. 347 Brazed with a Ni-Cr-Si-B-C Alloy.'

Welding Jnl., 1958, vol. 37, Mar., pp. 125S–8S.

Mechanical-property tests on high-temperature-alloy brazed joints have, until recently, been concerned mainly with determinations of tensile and shear strength: attention is directed in this paper to some major items of information. In order to obtain some idea of the effects of dynamic stresses on the interfaces of brazed joints, the present authors carried out a series of fatigue tests on brazed joints made in niobium-stabilized austenitic steel of the A.I.S.I. 347 type (carbon 0·08 max., nickel 9–12, chromium 17–19, per cent.), using a brazing alloy containing nickel 65–75, chromium 13–20, boron 2·75–4·75, iron+silicon+carbon 10, per cent. (*S.A.E./A.M.S.* 4775).

The single-lap-joint fatigue-test specimen used is illustrated and experimental conditions are described in detail. Brazing was carried out in a dry-hydrogen atmosphere of −60°F. (−51°C.) dew point.

The parent material, in the annealed condition, was found to have an endurance limit, at 50×10^6 cycles, of 31,600 p.s.i. (14 t.s.i.; 22 kg./mm.^{2}) at room temperature and 31,000 p.s.i. (13·8 t.s.i.; 21·8 kg./mm.^{2}) at 1100°F. (593°C.). At room temperature the endurance limit of the brazed joints was equivalent to that of the parent material, but at 1100°F. (593°C.) the value for the joints was about 10 per cent. lower than that of the parent steel.

Fatigue cracking in the flexed single-lap joint specimens occurred near the location of maximum change in stress, in the region immediately adjacent to the joint rather than through the joint interface. A mechanism is proposed to explain fatigue cracking in the flexed joints.

PATENTS

Electrorefining of Nickel: New Inco Process

The invention relates to the electrolytic refining, by means of an aqueous acidic sulphate-chloride electrolyte, of high-purity nickel, and production of elemental sulphur, from nickel-matte anodes containing between about 20-32 per cent. sulphur in the form of sulphides. The process covered by the patent has been described *in extenso* in a paper by RENZONI, MCQUIRE and BARKER: see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 6, pp. 155-6.

L. S. RENZONI and W. V. BARKER, assignors to INTERNATIONAL NICKEL CO. OF CANADA, LTD.
Canad. Pat. 556,649.

Separation of Nickel and Cobalt present in Aqueous Solution

The pH of solutions containing salts of nickel and cobalt is adjusted to about 2, and the solution is treated, at room temperature, with gaseous chlorine in combination with one or more alkaline- and/or alkaline-earth-metal carbonates. Additions of the gas and carbonate(s) are controlled so that the pH rises to, and is maintained at, approximately 3 until the greater part of cobalt present is precipitated as its hydroxide. The pH is then allowed to rise to 4, to ensure complete precipitation of the cobalt, and finally the cobalt hydroxide is filtered off and washed with water. Results of a typical separation showed a cobalt yield of 99·792 per cent.

STÉ. D'ÉLECTRO-CHIMIE, D'ÉLECTRO-MÉTALLURGIE ET DES ACIÉRIES ÉLECTRIQUES D'UGINE. *Brit. Pat.* 795,410.

Use of Nickel in Minimizing Clearances between Rotor-Blade Tips and Stator Casings in Gas Turbines

Clearances between the rotor-blade tips and the stator casing of axial-flow turbines and compressors at service temperatures are minimized by providing the inner surface of the casing opposite each row of blades with a cylindrical track made of porous sintered metal which is abraded by the rotor-blade tips during operation.

The patent claim covers also methods of forming the porous-metal track.

The preferred metal powder is nickel, which is applied to the copper-flashed stainless-steel stator surface and sintered in a non-oxidizing atmosphere at 1650°-1700°F. (900°-920°C.). Alternatively, a strong bond is achieved by using nickel-powder particles electrolessly plated with nickel phosphide, and using, instead of a copper flash, a coating of the nickel-phosphide eutectic deposited by electroless procedure on the stator surface. Other metals (iron, copper, silver, chromium or nickel-iron alloys) may be substituted for nickel, but are less satisfactory. Methods of sintering the nickel powder onto the assembled stator casing are described and illustrated.

GENERAL MOTORS CORPN., assignee of R. P. KOEHRING.
Brit. Pat. 794,947.

Bright-Nickel Plating Solutions

A series of three patents relates to the electrodeposition of fine-grained bright-nickel coatings from aqueous acidic solutions containing:

(*a*) one or more of the following salts: nickel sulphate, nickel chloride, nickel fluoborate, nickel sulphamate;

and

(*b*) one of the following additions:

(1) an alkyne sulphonic acid having 3-14 carbon atoms (preferably 2-butyne-1, 4-disulphonic acid; 2-butynoxy-1,4-di-(ethane sulphonic) acid or propargyl sulphonic acid; or, in conjunction with an organic sulphon compound, 2-propynoxy-1-ethane sulphonic acid or 1,1-dimethyl-2-propynoxy-1-ethane sulphonic acid) (*Patent No.* 2,800,440);

or

(2) about 0·005-2 g./L. of at least one alkynoxy alkane carboxylic acid having a total of 5-15 carbon atoms (preferably 2-butynoxyl-1,4-diacetic acid or 2-propynoxy-1-acetic acid), in conjunction with 0·1 g./L. to saturation of a sulphur-containing brightener such as diphenyl or naphthalene sulphonic acids (*Patent No.* 2,800, 441);

or

(3) about 0·005-4 g./L. of an unsaturated sulphonic acid having 3-14 aliphatic carbon atoms (preferably propynoxy phenyl sulphonic acid; 2-butyn-1,4-di(oxyphenyl sulphonic) acid; phenyl butynoxy sulphonic acid; or 2-sulphophenyl butynol), with or without an organic sulphon compound. (*Patent No.* 2,800,442).

Listed in each patent are examples of typical solutions and operating conditions, and preferred concentrations and combinations of addition agents.

H. BROWN and R. J. CLAUSS, assignors to UDYLITE RESEARCH CORPN. *U.S. Pats.* 2,800,440, 2,800,441, 2,800,442.

Reduction of 'Skip-Plating' Defects in Nickel Electrodeposits

The addition of 0·005-0·5 (preferably 0·01-0·2) g./L. of thiomalic (mercapto-succinic) acid to a Watts nickel-plating solution reduces the tendency for 'skip plating' and, in the presence of an organic brightener, extends the bright-plating capacity of the electrolyte to very low current densities. Particularly satisfactory results are claimed to have been achieved by addition of thiomalic acid in conjunction with a strong brightener of the water-soluble acetylenic type and/or a sulphon-oxygen compound. Examples of electrolytes of suitable composition are listed.

D. G. FOULKE and O. KARDOS, assignors to HANSON-VAN WINKLE-MUNNING CO. *U.S. Pat.* 2,818,376.

Electroless Deposition of Vanadium-Nickel Alloys

Binary alloys of vanadium with nickel, iron, cobalt or chromium are deposited by electroless technique from an aqueous plating solution containing a vanadium salt, a salt of the metal which is to be co-deposited, a buffering agent, and at least one reducing and complexing agent which maintains the positive ions of these salts in states of lowest ionic valence and simultaneously forms with them a complex which is readily reducible to the desired alloy. Finally, a hypophosphite salt is added (together with a suitable buffering agent), the pH is adjusted to a selected range (either acidic or basic) and the metal substrate is inserted into the solution. The complex formed on the substrate surface is then rapidly reduced to the alloy through the action of the hypophosphite ion. (It is stated that any metal which can be electrolessly plated with chromium may be plated by this method.)

Among the examples of suitable solutions cited in the patent is the following, for deposition of an 85–15 nickel-vanadium alloy:

	g./L.
$NiSO_4.7H_2O$	30
Sodium acetate	10
Sodium hypophosphite ..	10
$K_2C_2O_4.H_2O$	50
$NaVO_3$	5

P. H. EISENBERG and D. O. RALEIGH, assignors to SYLVANIA ELECTRIC PRODUCTS, INC.
U.S. Pat. 2,828,227.

Nickel-Copper-base Casting Alloy for Glass Moulds

The composition of an alloy claimed to show improved castability, fine grain-size, and high resistance to thermal shock and to corrosion and adhesion by molten glass, is within the following range: carbon 0·2 max., silicon 0·2–1·5, manganese 0·5–2·5, magnesium 0·02–0·1, copper 20–40, aluminium 0·5–17, titanium 0·01–0·5, boron 0·03–0·5, per cent., remainder nickel. The properties claimed make the alloy eminently suitable as a material of construction for moulds used in casting glass.

The alloys are produced by a treatment (also claimed in the patent) which involves oxidation by means of 0·25–0·35 per cent. of nickel oxide added to a molten bath containing nickel and 20–40 per cent. of copper, deoxidizing with specified amounts of silicon and manganese, carburizing with 0·1 to 0·2 per cent. of carbon, adding aluminium to give half the necessary final content, adding titanium in the required amount, then the remainder of the aluminium, boron (0·06 — 0·1 per cent.) and finally up to 0·05 per cent. of magnesium.

G. L. LEE, assignor to INTERNATIONAL NICKEL CO., INC.
U.S. Pat. 2,833,646.

Nickel-Boron-Copper Alloy for Nuclear-Reactor Control Rods

The alloy, claimed to have a high cross-section neutron-absorption ratio, a relatively short half-life, a high melting point, and satisfactory oxidation-resistance, ductility, shock-resistance and machinability, consists of 5–20 per cent. of boron, 25–79 per cent. of nickel and/or chromium, remainder copper.

L. V. LAROU, assignor to WALL COLMONOY CORPN.
Canad. Pat. 554,010.

Refractory Metal-Boride Tool Materials

The invention relates to hard refractory metal-boride compositions claimed to exhibit properties similar to, and in applications such as tool tips or hard-facing to offer advantages over tungsten carbide. Such boride compositions may be produced by compacting and sintering mixtures of powders of Mo_2NiB_2 and Mo_2B so as to form a eutectic phase between the two constituents: the eutectic, the ternary boride and molybdenum boride are in proportions corresponding to a composition range of boron 15–60, molybdenum 30–70, nickel 1–40, at. per cent. Examples are given to illustrate the preparation of the refractory compositions and the ratios used.

In the ternary boride the nickel may be replaced by cobalt.

AMERICAN ELECTRIC METAL CORPN.
Brit. Pat. 790,917.

Tarnishing of Nickel Silver and Other Copper-Nickel-containing Alloys by Detergents: Inhibitor Solutions

Detergents containing polyphosphates have in recent years found numerous domestic and other applications. Aqueous solutions of polyphosphates tend, however, at certain pH values, especially at elevated temperatures, to tarnish nickel silver, a material frequently used for household articles which might be washed in water containing such detergents.

The seven patents to which attention is drawn here cover akali-metal-phosphate detergent compositions claimed to obviate this deleterious effect. The inhibitors and the patents relating to them are noted below.

U.S. Pat. 2,829,103	A water-soluble inorganic salt of antimony or bismuth.
U.S. Pat. 2,829,104	Vanadyl sulphate.
U.S. Pat. 2,829,105	A water-soluble inorganic niobate.
U.S. Pat. 2,829,106	A water-soluble inorganic tantalate.
U.S. Pat. 2,829,107	A water-soluble ferrous salt.
U.S. Pat. 2,829,108	A water-soluble inorganic hydroxylamine salt in conjunction with an alkali-metal metasilicate.
U.S. Pat. 2,829,109	A water-soluble inorganic hydrazine salt.

Inhibitors covered by the last two patents are claimed to be also highly effective in minimizing detergent tarnishing of copper-nickel alloys of such types as coinage materials, cupro-nickel and 'Monel'.

Each of the patents includes particulars of preferred materials and examples of detergent compositions.

E. E. RUFF and E. E. SMITH, assignors to LEVER BROTHERS CO. *U.S. Pats.* 2,829,103-9.

Electrode for Welding Iron-containing Copper-Nickel Alloys

The patent relates to a flux-coated electrode claimed to produce sound, strong, pore-free, ductile arc-weld deposits on iron-bearing copper-nickel alloys of the 90-10 type. The composition of the core wire is in the following range: silicon 0·2-1, manganese 0·05-1, magnesium 0·08 max., nickel 5-15, iron 0·5-2, per cent., remainder copper. The dry flux is composed of calcium carbonate 6-26, calcium fluoride 15-30, cryolite 15-30, titania 20-30, manganese 1·7-8·5, calcium silicate 2-10, bentonite 2-5, per cent. Preferred compositions are detailed for both core and coating.

The patent includes examples illustrating compositions of weld-metal deposits from such electrodes and the properties obtainable in the weldments.

G. R. PEASE and T. E. KIHLGREN, assignors to INTERNATIONAL NICKEL CO., INC. *U.S. Pat.* 2,834,098.

Nickel-Iron Glass-to-Metal Sealing Alloy

The alloy '4750' (composition range nickel 47-49, iron 50, aluminium, silicon, manganese or chromium 1-3, per cent.) has a linear coefficient of thermal expansion, over the range 0°-300°C., similar to that of lead-borosilicate plate and window glass. Advantage is taken of this characteristic in the multiple-glazing industry (in applications, for example, such as metal spacers between the sheets of glass of a multiple-glazed unit). An effective seal is achieved between the alloy and a lime-soda-silica glass by means of a lead-borosilicate glass consisting of PbO 67-75, B_2O_3 12-16, SiO_2 10-15, Al_2O_3 0-5, per cent.

PITTSBURGH PLATE GLASS CO. *Brit. Pat.* 791,969.

High-Temperature Alloys containing Cerium

Additions of up to 1 per cent. of cerium to alloys within the composition limits cited in *Brit. Pat.* 746,472 are claimed to give improved properties, particularly with respect to rupture strength at high temperatures and forgeability. The cerium may be added in the form of ferrocerium, *or* Mischmetall, *or* mixed rare-earth oxides in conjunction with a suitable deoxidant by which they will be reduced, *or* any cerium-rich alloy.

The alloys are of the following preferred percentage composition: carbon 0·15-0·45; manganese 0·5-1·5; silicon 0·15-0·75; chromium 15-23; nickel 13-28; cobalt 0 or 22-28 (nickel + cobalt 45 per cent. max.); boron 0·002-0·04; titanium, molybdenum, tungsten 0·5-3·5, respectively, the total being between 5 and 10; cerium 1 max.; remainder iron.

WILLIAM JESSOP AND SONS, LTD. (inventors, G. T. HARRIS and H. C. CHILD). *Brit. Pat.* 795,377 (addition to *Brit. Pat.* 746,472). Similar to *Belgian Pat.* 544,189.

Nickel-base-Alloy Turbine Blading with Improved Resistance to Thermal Shock

Greatly improved resistance to thermal shock is conferred on nickel- or cobalt-base-alloy turbine blading by decarburizing, and lowering the boron content of, a surface layer 0·001-0·01 in. (0·025-0·25 mm.) deep, a process which is accomplished by heating the alloy in a hydrogen atmosphere at a temperature in the range 2100°F. (1150°C.) to just below its melting point. Decarburization of such a very thin skin is claimed to have little effect on the stress-rupture properties. Alloys covered fall into the following composition range: carbon up to 1, chromium 10-30, molybdenum and/or tungsten 0-25, niobium and/or tantalum and/or titanium 0-8, iron up to 25, boron up to 0·5, per cent., balance nickel and/or cobalt.

F. S. BADGER, assignor to UNION CARBIDE AND CARBON CORPN. *Canad. Pat.* 549,445.

Austenitic Stainless Steels for High-Temperature Applications

This series of patents specifies composition limits for austenitic stainless steels intended as materials of construction for superheater tubing. The materials described have relatively low 'strategic-metal' content, and satisfy the following requirements: (1) stress-rupture strength at 1350°F. (732°C.) at least twice that of the straight 18-8 chromium-nickel steels; (2) adequate resistance, at 1350°F. (732°C.), to corrosion by superheated vapour and combustion gases; (3) adequate hot plasticity for fabrication into tubing; (4) favourable mechanical properties; (5) weldability; and (6) freedom from serious embrittlement at service temperatures.

The steels are of the following base composition: carbon 0·02-0·15, silicon 0·10-1·00, manganese 0·25-2·50, chromium 15-20, nickel 12-18, per cent. Alloying additions claimed substantially to increase the creep-rupture strength of steels of the base composition are noted below, correlated with the patent which covers their use.

Pat. 2,823,114	Niobium-tantalum 1-3·5, per cent.
Pat. 2,824,795	Niobium-tantalum 1-3·5, copper 2-3, molybdenum 1-2, per cent.
Pat. 2,824,796	Copper 2-3, molybdenum 2-3, tantalum 0·5-2, nitrogen 0·15-0·25, per cent.
Pat. 2,824,797	Copper 2-3, molybdenum 1-2, niobium-tantalum 0·5-2, vanadium 0·2-1, nitrogen 0·1-0·25, per cent.

Preferred compositions, and stress-rupture properties at 1350°F. (732°C.), are listed in each patent.

F. T. EBERLE and C. L. COREY, assignors to BABCOCK AND WILCOX CO. *U.S. Pats.* 2,823,114, 2,824,795, 2,824,796, 2,824,797.

Nickel-Alloy Multiple-Thermocouple Elements

In a thermocouple designed to permit determination of temperatures at several points in rapid succession or simultaneously, an elongated tube, preferably of a nickel-chromium alloy, comprises a common element in a series of thermo-electric junctions, the other elements of which consist of nickel-aluminium alloy wires attached at intervals along the length of the tube and each forming a separate circuit. By arrangement of the circuits in series, temperature can thus be determined at several points, or, by means of a selector switch, measurements can be made in rapid succession. The application of the device to determine the coefficient of thermal expansion of glass is described.

GENERAL ELECTRIC CO., LTD. (inventors, W. S. LEE and D. G. S. M. OSBORNE). *Brit. Pat.* 795,035.

High-Temperature Nickel-containing Cermets

The patent relates to cermet compositions claimed to exhibit high strength and heat-resistance at temperatures up to 1000°C. The materials are produced by powder-metallurgy methods, from compacts consisting of particles each of which contains

(*a*) 0·5–25 per cent. of a refractory material of high melting point, such as an oxide of calcium, beryllium, magnesium, etc. (which would act as a hardening constituent and as a recovery-inhibiting phase);

(*b*) a metal of relatively high melting point and of a ductile type, for example, a heat-resisting alloy.

The size of the composite powder particles, in which one constituent should be substantially covered by the other and both should be non-segregable, is limited to 40μ (preferably 5μ). The thickness of the metallic coating on the refractory material is 0·05–1μ and, in particles where the reverse procedure is used, the refractory coating should be up to 0·3μ thick. Details are given of techniques which may be employed to produce the powders in the desired form. The preferred metallic constituent is an alloy of the following percentage composition: chromium 5–30, iron 25 max., nickel + cobalt remainder (preferably 40–50 per cent. of the total). Production is completed by sintering the compact at a temperature in the region of 1000°C. and finally hot-working, to achieve a reduction of at least 50 per cent., as a means of eliminating voids.

N. J. GRANT and C. G. GOETZEL, assignors to SINTERCAST CORPN. OF AMERICA. *U.S. Pat.* 2,823,988.

Heat-Resisting Titanium-Carbide/Nickel-Alloy Compositions

Heat-, corrosion- or abrasion-resisting components, such as turbine blades, valves and tools, are produced by heating a porous, shaped skeleton of a refractory-metal compound (e.g., of carbides, borides, nitrides and/or silicides of titanium, zirconium, chromium, molybdenum, tungsten, vanadium, niobium or tantalum) in the presence of a molten matrix-forming metal or alloy, so as to ensure the infiltration of the latter throughout the refractory skeleton. Zirconia, containing additions of crystallographic modifiers to produce a structure at least 95 per cent. cubic, is claimed as a non-reactive refractory support or mould for the porous skeleton during the operation. Examples of suitable nickel-, cobalt- or iron-base matrix-forming alloys are given. Compositions of nickel base include: 80–20 nickel-chromium; 80–14–6 nickel - chromium - iron ; and complex nickel - chromium-iron and nickel-chromium-molybdenum alloys.

C. G. GOETZEL and L. P. SKOLNICK, assignors to SINTERCAST CORPN. OF AMERICA. *U.S. Pat* 2,828,226.

Nickel-Chromium-Cobalt-Molybdenum Valve-Seat Inserts

The patent relates to valve-seat inserts of the type employed in internal-combustion engines. Substantially increased resistance to 'collapse', valve-lash changes and corrosion, together with excellent hot strength and little tendency to attract deleterious deposits from the products of combustion, is obtained in valves cast from an alloy within the following composition range: carbon 2·25-2·75 (2·5), nickel 35–40 (37·5), chromium 32–38 (35), cobalt 10–14 (12), molybdenum 6–8 (7), iron 8 max. (6), per cent. (The preferred composition is shown in brackets.) The alloy is 'cast and cooled to provide a cast structure including dendrites dispersed in a solid solution of a eutectic matrix.'

H. F. PRASSE and A. A. ARMSTRONG, assignors to THOMPSON PRODUCTS, INC. *U.S. Pat.* 2,827,373.

Precipitation-Hardenable Chromium-Nickel-Phosphorus-Copper Stainless Steel

A low-carbon stainless steel of the composition shown below is, by virtue of controlled additions of copper and phosphorus, age-hardenable from the hot-worked condition without need for prior high-temperature solution treatment. Composition limits (per cent.) are given as carbon 0·1–0·3, phosphorus 0·15–0·4, copper 0·5-about 3, chromium 12–30 (approx.), nickel + manganese 3–35 (nickel at least 3). Boron (0·002–0·01 per cent.) and also nitrogen (at least 0·15 per cent.) may also be present.

Heat-treatment involves hot working followed by age-hardening at 1200°–1500°F. (650°–815°C.), to achieve a hardness value of at least Rockwell C 30. Hardness effects obtainable in individual steels within the broad range are tabulated.

W. R. KEGERISE, assignor to CARPENTER STEEL CO. *U.S. Pat.* 2,826,496.

Precipitation-Hardenable Stainless Steels and their Heat-Treatments

The patent claims precipitation-hardenable corrosion-resisting steels within the percentage limits shown below: preferred limits are indicated in brackets. Carbon up to 0·20 (0·05–0·12), silicon up to 2·00 (0·2–1·0), manganese up to 5·0 (0·2–4·0), chromium 12·0–21·0 (16–19), nickel up to 11·5 (4–7),

nitrogen up to 0·20 (0·01–0·10). Emphasis is placed on correct balancing of chromium and silicon on the one hand and nickel, carbon, manganese and nitrogen on the other: the significance of this aspect is demonstrated by reference to the properties of steels in which the proportions of the elements are varied. Steels according to the invention are claimed to be ductile and readily workable and to have a high strength/weight ratio.

Heat-treatment involves annealing in the range 1700–2200°F. (925–1205°C.), cooling, re-heating to between 1200° and 1700°F. (650°–925°C.), cooling, and hardening in the range 300°–1150°F. (150°–620°C.). Preferred heat-treatments are cited for the steel in the cast or wrought conditions, together with the hardness and mechanical properties which they achieve.

J. H. WAXWEILER, assignor to ARMCO STEEL CORPN.
U.S. Pat. 2,820,708.

Heat-Treatment of Stainless-Steel Articles with Cutting Edges

Articles such as surgical blades, pins and needles, which are fabricated from stainless steel to obtain the advantage of corrosion-resistance, are given keener and sharper cutting edges or points than have previously been attainable in such material by heating, after grinding, to a temperature within the range 265°–270°C., slowly increasing the temperature to the region of 285°C. and finally cooling to room temperature. If necessary, further finish-grinding may be carried out after this thermal treatment.

The patent covers also the application of this treatment to the manufacture of a surgical needle from an 18–8-type stainless steel of the following composition range: carbon 0·1 max., manganese 0·4–0·7, silicon 0·45–0·75, chromium 17–19, nickel 8–9·5, per cent.

STAINLESS SURGICAL NEEDLE CO., LTD.
(inventors, W. D. SMALLWOOD and C. A. PAYNE).
Brit. Pat. 791,113.

Permeable Sintered Chromium-Nickel Stainless-Steel Products

Stainless-steel articles of uniform permeability, characterized by corrosion-resistance and by a good degree of strength at relatively high temperatures, are produced by powder-metallurgy methods, using additions of carbon to widen the solidus and liquidus range of the steel sufficiently to permit adequate furnace control and to lower the melting point of the mass, without damaging the desirable properties of the steel. The carbon should be added to the stainless-steel powder in amounts of 0·5 to 1·25 per cent. The powder mixture is loose-sintered, and decarburized in a hydrogen atmosphere (within the solidus-liquidus range either simultaneously with, or after, sintering), bringing the carbon content to a level normal in such steels, thus ensuring good corrosion-resistance in the finished permeable product. Materials made according to this invention are suitable for filters and other articles in which a controlled degree of porosity is required.

R. L. PROBST and G. J. LE BRASSE, assignors to FEDERAL-MOGUL CORPN. *U.S. Pat.* 2,826,805.

Production of Ketene in Stainless-Steel Reactors

Ketene is obtained in high yields by the vapour-phase catalytic pyrolysis of acetic acid at pressures ranging from atmospheric to about 20 lb./sq. in. and at a temperature between 625° and 750°C. The material of construction used for the reactor is of vital importance, since many metals will, under the operating conditions, convert the ketene into unwanted substances, or will fail to withstand the high temperatures involved. Steels claimed as suitable for this purpose are those which lie within the compositional limits chromium 17–20, nickel 8–12, molybdenum 3–4, per cent.

O. V. LUKE, M. O. ROBESON and W. E. TAYLOR, assignors to CELANESE CORPN. OF AMERICA.
U.S. Pat. 2,820,058.

Nickel-base High-Phosphorus Brazing Materials

Corrosion-resistant, ductile bonds are achieved between such materials as cast iron, cast steels, titanium alloys, molybdenum and stainless steels by applying to the joints a paint-like mixture consisting of water + an ammonium phosphate (preferably $(NH_4)_2HPO_4$ or $(NH_4)\ H_2PO_4$) + at least one oxide of nickel, copper, molybdenum, tungsten and iron (preferably NiO and/or CoO, or NiO and CuO or NiO and WO_3), and finally heating the coated joints in a reducing atmosphere, so that the oxides are converted to metals which alloy and act as the brazing material. Numerous typical examples are cited.

G. J. HORVITZ, assignor to NIPHOS CORPN.
U.S. Pat. 2,822,609.

Titanium-Nickel-base Brazing Alloys

Self-fluxing brazing powders suitable for bonding titanium or titanium-base alloys to other metals or alloys, for example in applications such as gas-turbine compressor blading, consist essentially of the eutectic formed of 28 per cent. nickel and 72 per cent. titanium. Additions of copper and/or silver are claimed as a means of lowering the melting point of the braze and so decreasing grain growth, while small additions of one or more metals such as chromium, iron, manganese and cobalt are stated to have a beneficial effect on the properties of the final joint. Composition limits are defined as: titanium-nickel eutectic 65–95, copper and/or silver 5–35, iron, chromium, etc., 2–15, per cent. From trace amounts up to 6 per cent. of powdered titanium may also be present. Typical compositions are listed, with particulars of the melting points of the respective alloys.

R. A. LONG. *U.S. Pat.* 2,822,269.

Laminated Nickel-Chromium Electrode

Metal electric-resistance brazing electrodes are fabricated from longitudinal laminations, the resiliency of which permits the contact surface to conform more closely to the work surface than would that of a solid electrode, thus reducing the risk of sparking and considerably increasing the life of the electrode. Nickel-chromium alloys having a specific resistance of 108 microhm/cm.^{3} are stated to be eminently suitable for such electrodes.

BRITISH THOMSON-HOUSTON CO., LTD. (inventor, F. M. SHARMAN). *Brit. Pat.* 791,921.

Heat-Treatment and Brazing of Oxidizable Alloys

A component or alloy containing a readily oxidizable metal is satisfactorily heat-treated to produce or retain a bright surface finish by effecting the treatment *in vacuo*, preferably at a pressure of less than 50 microns of mercury (and, for optimum results, of one micron or less), in the presence of, but not in contact with, a carbonaceous material (e.g., graphite heating elements) which completes the removal of oxygen from the furnace atmosphere. The technique is applicable also to brazing readily oxidizable articles, to the use of brazing alloys which have proved difficult to deposit satisfactorily in the reducing atmospheres usually employed, and to sintering of powder compacts.

The patent gives details of the following specific applications of the invention:

- Brazing fins of nickel-chromium-base alloy to nickel tubing (brazing alloy 80-20 copper-nickel);
- Brazing nickel-cobalt-iron alloy (29-17-54% type) to mild steel (brazing alloy 'Nicrobraz');
- Brazing nickel-chromium-cobalt-base alloy to similar material (brazing alloy 'Nicrobraz');
- Brazing 18-8 titanium-stabilized chromium-nickel steel to similar material (brazing alloy 'Nicrobraz');
- Sintering of powder compacts of nickel-chromium-cobalt-base alloy.

GENERAL ELECTRIC CO., LTD. (inventors, H. D. BLAKELOCK and R. G. COFFIN). *Brit. Pat.* 793,251.

Welding Rod for Deposition of Titanium-stabilized Weld Metal

The patent covers the use (on titanium-stabilized 18-8-type stainless-steel welding rods) of an electrode-coating composition which results in production of titanium-stabilized arc-weld deposits. The coating contains silicates in an amount not exceeding five times the combined weight of the titanium and aluminium content, and not more than 1 per cent. by weight of reducible carbon compounds, in such proportions as to produce weld metal with a silicon content of not more than 1 per cent. and a residual titanium content of not less than five times the carbon content. The preferred composition is cited as: calcium 15-35, aluminium oxide 1-8, titanium dioxide 30-60, clay 1-5, manganese 1-7, magnesium + calcium oxide 1-10, ferro-chromium 1-10, titanium-aluminium alloy 1-20, per cent., silicate binders not exceeding 5 times the titanium-aluminium alloy.

Data are presented to illustrate the satisfactory mechanical properties and corrosion-resistance of weld deposits made with rods so coated.

L. M. KEE, assignor to AIR REDUCTION CO., INC. *U.S. Pat.* 2,825, 793.

Alternating-Current Arc Welding of Stainless and Mild Steel

An improved alternating-current arc-welding process makes use of an arc-shielding gas mixture of oxygen + argon (or helium) disposed around a fusible metal electrode, and of a high-frequency alternating potential superimposed on the alternating welding current. The welding current is of an intensity such that, in conjunction with the shielding gas and high-frequency potential, it results in the weld metal being deposited in the form of a spray, with stable operation, improved weld contour and at a lower alternating current level than was previously possible. The gas mixture preferably consists of 5 per cent. oxygen and 95 per cent. argon.

The patent includes details of procedure and conditions suitable for welding mild steel and chromium-nickel stainless steel.

UNION CARBIDE CORPN. *Brit. Pat.* 791,176.

Nickel-base Hard-Facing Alloy

A hard-surfacing alloy of the composition shown below is claimed to be especially suitable for deposition by means of brazing and high-speed spraying. It is stated to have good wetting and flow characteristics and corrosion-resistance, exceptionally high resistance to fretting corrosion, and to be particularly suitable for high-temperature applications. Composition limits: carbon 0·05-2·5, chromium 7-17, boron 1-4·5, silicon 1-5·5, iron 0·1-5·5, tungsten and/or molybdenum 6-20, per cent., balance nickel. Preferred and typical compositions are given.

WALL COLMONOY CORPN. (inventor, H. S. GONSER). *Brit. Pat.* 791,229.

Corrigendum

Nickel Bulletin, 1958, vol. 31, No. 5, p. 135.
'Cold Rolling Mill for Very Thin Strip.'
For *Engineering*, 1958, vol. 205, Feb. 21, pp. 272-4 read *Engineer*, 1958, vol. 205, Feb. 21, pp. 272-4.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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